

# EAST Search History

10/536, 492

Ref #	Hits	Search Query	DBs	Default Operator	Plurals	Time Stamp
L1	1562	544/405	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT	OR	ON	2007/03/11 13:55
L2	✓ 16	l1 and (oltipraz or dithiolane or 1, 2-dithiolane or 1,2-dithiol or dithiole or 1,2-dithiole)	US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT	OR	ON	2007/03/11 14:55
L3	— 1	("7012148").PN.	USPAT	OR	OFF	2007/03/11 14:49
L4	3	"2002026909"	EPO; JPO; DERWENT	OR	OFF	2007/03/11 14:50
L5	2	"02026909"	EPO; JPO; DERWENT	OR	OFF	2007/03/11 14:50
L6	↙ 2	CURPHEY.IN.	EPO; JPO; DERWENT	OR	OFF	2007/03/11 14:50
L7	1	("20030191137").PN.	US-PGPUB	OR	OFF	2007/03/11 14:55

# STN SEARCH TRANSCRIPT

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10/536,492

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NEWS	5	NOV 03	JAPIO enhanced with IPC 8 features and functionality
NEWS	6	NOV 10	CA/CAPLUS F-Term thesaurus enhanced
NEWS	7	NOV 10	STN Express with Discover! free maintenance release Version 8.01c now available
NEWS	8	NOV 20	CA/CAPLUS to MARPAT accession number crossover limit increased to 50,000
NEWS	9	DEC 01	CAS REGISTRY updated with new ambiguity codes
NEWS	10	DEC 11	CAS REGISTRY chemical nomenclature enhanced
NEWS	11	DEC 14	WPIDS/WPINDEX/WPIX manual codes updated
NEWS	12	DEC 14	GBFULL and FRFULL enhanced with IPC 8 features and functionality
NEWS	13	DEC 18	CA/CAPLUS pre-1967 chemical substance index entries enhanced with preparation role
NEWS	14	DEC 18	CA/CAPLUS patent kind codes updated
NEWS	15	DEC 18	MARPAT to CA/CAPLUS accession number crossover limit increased to 50,000
NEWS	16	DEC 18	MEDLINE updated in preparation for 2007 reload
NEWS	17	DEC 27	CA/CAPLUS enhanced with more pre-1907 records
NEWS	18	JAN 08	CHEMLIST enhanced with New Zealand Inventory of Chemicals
NEWS	19	JAN 16	CA/CAPLUS Company Name Thesaurus enhanced and reloaded
NEWS	20	JAN 16	IPC version 2007.01 thesaurus available on STN
NEWS	21	JAN 16	WPIDS/WPINDEX/WPIX enhanced with IPC 8 reclassification data
NEWS	22	JAN 22	CA/CAPLUS updated with revised CAS roles
NEWS	23	JAN 22	CA/CAPLUS enhanced with patent applications from India
NEWS	24	JAN 29	PHAR reloaded with new search and display fields
NEWS	25	JAN 29	CAS Registry Number crossover limit increased to 300,000 in multiple databases
NEWS	26	FEB 13	CASREACT coverage to be extended
NEWS	27	Feb 15	PATDPASPC enhanced with Drug Approval numbers
NEWS	28	Feb 15	RUSSIAPAT enhanced with pre-1994 records
NEWS	29	Feb 23	KOREAPAT enhanced with IPC 8 features and functionality
NEWS	30	Feb 26	MEDLINE reloaded with enhancements
NEWS	31	Feb 26	EMBASE enhanced with Clinical Trial Number field
NEWS	32	Feb 26	TOXCENTER enhanced with reloaded MEDLINE
NEWS	33	Feb 26	IFICDB/IFIPAT/IFIUDB reloaded with enhancements
NEWS	34	Feb 26	CAS Registry Number crossover limit increased from 10,000 to 300,000 in multiple databases

NEWS EXPRESS NOVEMBER 10 CURRENT WINDOWS VERSION IS V8.01c, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 25 SEPTEMBER 2006.

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FILE LAST UPDATED: 9 Mar 2007 (20070309/ED)

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=> S XYLENE AND TOLUENE AND IMPURITY

109467 XYLENE

7572 XYLENES

111379 XYLENE

(XYLENE OR XYLENES)

172318 TOLUENE

1526 TOLUENES

172885 TOLUENE

(TOLUENE OR TOLUENES)

164247 IMPURITY

203663 IMPURITIES

304455 IMPURITY

(IMPURITY OR IMPURITIES)

L1 377 XYLENE AND TOLUENE AND IMPURITY

=> S L1 AND TRACES OF XYLEN?

75330 TRACES

126756 XYLEN?

17 TRACES OF XYLEN?

(TRACES(1W)XYLEN?)

L2 0 L1 AND TRACES OF XYLEN?

=> S L1 AND %

0 %

L3 0 L1 AND %

=> S L1 AND COMMERCIAL TOLUENE

34036 COMMERCIAL

31 COMMERCIALS

34066 COMMERCIAL

(COMMERCIAL OR COMMERCIALS)

312939 COM

77 COMS

313000 COM

(COM OR COMS)

329631 COMMERCIAL

(COMMERCIAL OR COM)

172318 TOLUENE

1526 TOLUENES

172885 TOLUENE

(TOLUENE OR TOLUENES)

28 COMMERCIAL TOLUENE

(COMMERCIAL(W)TOLUENE)

L4 0 L1 AND COMMERCIAL TOLUENE

=> S L1 AND TECHNICAL

33478 TECHNICAL

5 TECHNICALS

33482 TECHNICAL

(TECHNICAL OR TECHNICALS)

100702 TECH

124298 TECHNICAL

(TECHNICAL OR TECH)

L5 5 L1 AND TECHNICAL

=> D 1-5 IBIB ABS

L5 ANSWER 1 OF 5 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2006:485343 CAPLUS

DOCUMENT NUMBER: 145:34410

TITLE: Use of Mass Spectrometry Methods as a Strategy for Detection and Determination of Residual Solvents in Pharmaceutical Products

AUTHOR(S): Perez Pavon, Jose Luis; del Nogal Sanchez, Miguel; Garcia Pinto, Carmelo; Fernandez Laespada, M. Esther; Moreno Cordero, Bernardo

CORPORATE SOURCE: Departamento de Quimica Analitica Nutricion y Bromatologia, Facultad de Ciencias Quimicas, Universidad de Salamanca, Salamanca, 37008, Spain

SOURCE: Analytical Chemistry (2006), 78(14), 4901-4908

CODEN: ANCHAM; ISSN: 0003-2700

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB In the present work a strategy for the qual. and quant. anal. of residual solvents in pharmaceutical products is reported. First, a low-resolution chromatogram is generated for the identification of the solvents present in the samples by means of headspace generation-fast gas chromatog.-mass spectrometry (HS-fast GC/MS). From the plotting of this information by means of contour plots with time and mass/charge axes, it is decided whether quantification of such compds. can be accomplished without chromatog. separation or whether it should be done by fast gas chromatog. The nonseparative method is based on direct coupling of a headspace sampler with a mass spectrometer (HS-MS) and requires a signal recording time of only 3 min, while with fast gas chromatog. the time required to obtain a

chromatogram is 7.16 min. The use of headspace generation for introducing the sample and standard addition as a quantification technique provided satisfactory results and minimized the matrix effect. An important advantage of the methodologies used here is related to the fact that no prior treatment of the sample is required, thus minimizing the creation of anal. artifacts and the errors associated with this step of the anal. process. The methods were applied to the determination of residual solvents

in 27

different pharmaceutical products. Detection and quantitation limits were sufficiently low to enable the estimation of organic volatile impurities according to the International Conference on Harmonization (ICH) of Tech. Requirements for the Registration of Pharmaceuticals for Human Use.

REFERENCE COUNT: 24 THERE ARE 24 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L5 ANSWER 2 OF 5 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2002:961709 CAPLUS

DOCUMENT NUMBER: 138:44811

TITLE: Analysis of residual solvents in pharmaceuticals by headspace gas chromatography

AUTHOR(S): Nagai, Yumi

CORPORATE SOURCE: Analytical Applications Department, Analytical & Measuring Instruments Division, Shimadzu Corporation, Kyoto, Japan

SOURCE: Shimadzu Hyoron (2002), 59(1/2), 57-67

CODEN: SHHYAG; ISSN: 0371-005X

PUBLISHER: Shimadzu Hyoron Henshubu

DOCUMENT TYPE: Journal

LANGUAGE: Japanese

AB It has been a general practice that the anal. of residual solvents in pharmaceuticals is carried out by dissolving the pharmaceutical sample in a suitable solvent and then injecting that solution directly into a GC (gas chromatograph). Headspace gas chromatog. has recently come to be widely used for this application. In this method, the sample solution is enclosed in a vial, which is then kept at an appropriate temperature to drive out the highly volatile components into the gas phase in the vial, and then the gas phase is analyzed by gas chromatog. This report refers to our studies of the resolution of the Class 1 and Class 2 solvents to according to the Impurities Guidelines adopted by the ICH (The International Conference on Harmonization of Tech. Requirements for Registration of Pharmaceuticals for Human Use), to the Headspace GC adopted in the USP (United States Pharmacopoeia) <467> titled Organic VOLATILE IMPURITIES, and also to our investigation about the relative sensitivities of the Class 1 and Class 2 solvents detected by an FID (flame ionization detector) and an ECD (electron capture detector), and about those dissolved by the different solvents [aqueous solution DMSO solution, and DMF (N,N-dimethylformamide) solution].

L5 ANSWER 3 OF 5 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1994:413786 CAPLUS

DOCUMENT NUMBER: 121:13786

TITLE: Pressurized fluidized-bed gasification experiments with wood, peat and coal at VTT in 1991-1992. Part 1. Test facilities and gasification experiments with sawdust

AUTHOR(S): Kurkela, Esa; Stahlberg, Pekka; Laatikainen, Jaana

CORPORATE SOURCE: Lab. Fuel and Process Technol., Finland

SOURCE: VTT Publications (1993), 161, 57PP

CODEN: VTTPEY; ISSN: 1235-0621

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Fluidized-bed air gasification of Finnish pine sawdust was studied in the PDU-scale test facilities of the Tech. Research Center of

Finland to support the development of simplified integrated gasification combined-cycle processes by providing new information on the formation and behavior of different gas impurities in wood gasification. The gasifier was operated at 4-5 par pressure and at 880-1020°. The product gas was cleaned by ceramic candle filters operated at 490-715°. Concns. of tars, fixed N species, and vapor-phase alkali metals were determined in different operating conditions. C conversion exceeded 95% in all test periods although the gasifier was operated without recycling the cyclone or filter fines back to the reactor.

L5 ANSWER 4 OF 5 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1956:67083 CAPLUS  
DOCUMENT NUMBER: 50:67083  
ORIGINAL REFERENCE NO.: 50:12479e-i  
TITLE: The linear crystallization of TNT systems  
AUTHOR(S): Gey, W. A.; Dalbey, E. R.; Van Dolah, R. W.  
CORPORATE SOURCE: U.S. Naval Ordnance Test Sta., China Lake, CA  
SOURCE: Journal of the American Chemical Society (1956), 78, 1803-10  
CODEN: JACSAT; ISSN: 0002-7863  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The linear crystallization velocity of tech. TNT at 74° was 1.56 cm./min.; that of the most highly purified sample (m. 81.5°) was 3.10 cm./min. When additives were used, the velocities for initially pure and tech. TNT were almost identical. The phase diagrams of the binary systems of TNT with 1-phenoxy-2-(2,4,6-trinitrophenoxy) ethane (I), 2,4,6-trinitrostilbene (II), and 2,3',4,6-tetranitrostilbene (III) all showed ideal solns. with a single eutectic. The TNT-I eutectic is at 67° and 25 moles % I; for TNT-II it is at 75° and 13 moles % II; for TNT-III the eutectic is at 77° and 8 moles % III. 2,4,6-Trinitrostilbene and its derivs. had the greatest effect in decreasing the crystallization velocity of TNT of all the additives studied.

The

trinitrophenyl group uniformly decreased linear crystallization, and an ethylene

bridge to the rest of the mol. created greater activity than -O-, -NH-NH-, -N:N-, -S-, -OC2H4O-, and -OC(CH3)2O-. The mechanism of retardation is considered to be adsorption of the impurity on the crystallizing TNT. A modified Langmuir isotherm equation was developed that did not account for the more efficient additives at low concentration; a more complete expression, accounting for irreversible adsorption of strongly adsorbed additives, was also developed. New compds. prepared for this study and their colors and m.ps. are: p-di(2,4,6-trinitrophenylethenyl)benzene, orange-yellow, 300°; 3'-iodo-2,4,6-trinitrostilbene, greenish yellow, 156-6.5°; 4'-phenyl-2,4,6-trinitrostilbene, orange, 223-3.5°; 2'-chloro-2,4,6-trinitrostilbene, greenish yellow, 145.5-6.0°; 4'-hydroxy-2,4,6-trinitrostilbene, bright-red, 219-20°; 2'-methoxy-2,4,6-trinitrostilbene, reddish orange, 151.5-2.0°; 2',4'-dichloro-2,4-dinitrostilbene, orange, 216-16.5°; 2'-hydroxy-2,4-dinitrostilbene, bright-yellow, 192.5-3.5°; 3'-methyl-2,4-dinitrostilbene, bright-yellow, 145-7°; 4'-phenyl-2,4-dinitrostilbene, bright-yellow, 178-9°; 4'-isopropyl-2,4-dinitrostilbene, bright-yellow, 179-9.5°; N-cetylpicramide, yellow, 85-5.5°; 1-phenoxy-2-(2,4,6-trinitrophenoxy)ethane, tan, 114-15°; 1-phenoxy-2-(2,4,6-trinitrophenoxy)-propane, tan, 118-19.5°; 2,4,6-trinitrodiphenyl sulfide, yellow, 122-3°; 2,4,6-trinitrophenyl-n-butyl ether, white, 37-8°; 2,4,6-trinitrophenyl-n-hexyl ether, white, 32.5-4°; 2-phenoxyethyl 3,5-dinitrobenzoate, white, 73.6-5°.

L5 ANSWER 5 OF 5 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1926:11280 CAPLUS  
DOCUMENT NUMBER: 20:11280

ORIGINAL REFERENCE NO.: 20:1395i,1396a-c  
TITLE: The effect of various purification methods on xylen as solvent for catalytic reductions  
AUTHOR(S): Zetzsche, Fritz; Arnd, Olga  
SOURCE: Helvetica Chimica Acta (1926), 9, 173-7  
CODEN: HCACAV; ISSN: 0018-019X  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The value of various hydrocarbons as solvents in the reduction of acid chlorides to aldehydes varies with their purity. The aldehyde-titer may be used as an index of their value. The impurities may (1) inactivate the catalyst; (2) allow a satisfactory reduction to aldehyde to take place; (3) the reduction goes past the aldehyde stage. To (1) belong most technical toluene, xylene, etc., and to (3) belong mostly the pure manufactured hydrocarbons. The latter class may be controlled by regulators. The reduction of BzCl was studied, the object being to convert a solvent of titer -0 into one of +0, and if possible to make the solvent inactive catalytically. Washing solvents, e. g., xylene, with concentrated H<sub>2</sub>SO<sub>4</sub> 20-25 times accomplishes this, but the loss is great. Distillation will not work. Treatment with POCl<sub>3</sub> seems quite satisfactory, although the solvent so obtained is not entirely inactive catalytically. Of a series of other substances with which xylene was treated to render it catalytically neutral (Na-Hg, Cu, C<sub>6</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>3</sub>OH, H<sub>3</sub>PO<sub>4</sub>, animal charcoal, HCl, AlCl<sub>3</sub>, and quinoline-S) only anhydrous AlCl<sub>3</sub> was satisfactory. One treatment with this is equivalent to 12 washings with concentrated H<sub>2</sub>SO<sub>4</sub>. Both POCl<sub>3</sub> and AlCl<sub>3</sub> give solvents for catalytic reductions to aldehydes which are usually satisfactory.

=> S L1 AND TRACES OF XYLENE

75330 TRACES  
109467 XYLENE  
7572 XYLENES  
111379 XYLENE  
(XYLENE OR XYLENES)  
12 TRACES OF XYLENE  
(TRACES (1W) XYLENE)

L6 0 L1 AND TRACES OF XYLENE

=> S TRACES OF XYLENE

75330 TRACES  
109467 XYLENE  
7572 XYLENES  
111379 XYLENE  
(XYLENE OR XYLENES)

L7 12 TRACES OF XYLENE  
(TRACES (1W) XYLENE)

=> D 1-12 IBIB ABS

L7 ANSWER 1 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1990:22726 CAPLUS

DOCUMENT NUMBER: 112:22726

TITLE: Hydrocracking of benzene in the presence of nickel zeolite catalysts obtained from bis(cyclopentadienyl)nickel

AUTHOR(S): Golubchenko, I. T.; Galich, P. N.; Motornyi, V. G.

CORPORATE SOURCE: Otd. Neftekhim., Inst. Fiz.-Org. Khim. Uglekhim., Kiev, USSR

SOURCE: Neftepererabotka i Neftekhimiya (Kiev) (1989), 36, 7-8

CODEN: NEFNBY; ISSN: 0548-1406

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB Complete transformation of benzene (I) to CH<sub>4</sub> was obtained in the presence of 3% Ni/NaX and 3% Ni/NaY zeolites at 400°, space velocity (liquid

I) 0.5 h<sup>-1</sup>, and H-I molar ratio 10. In the presence of catalysts containing 9.5% Ni, complete transformation of I was achieved at 390° and 1.0 h<sup>-1</sup>. At 3 h<sup>-1</sup> and 380° I conversion to CH<sub>4</sub> was 49-50.1% in the presence of a catalyst containing 3% Ni and 74.6-75.3% at 9.5% Ni. In the liquid products of incomplete conversion of I, appreciable amts. of toluene and traces of xylenes were present. The catalysts were obtained by calcining dehydrated zeolites impregnated with bis-(cyclopentadienyl)nickel.

L7 ANSWER 2 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1987:95277 CAPLUS  
DOCUMENT NUMBER: 106:95277  
TITLE: Multidimensional gas chromatography  
AUTHOR(S): Morishita, Fujio  
CORPORATE SOURCE: Coll. Eng., Kyoto Univ., Kyoto, Japan  
SOURCE: Bunseki (1986), (9), 618-25  
CODEN: BUNSD3; ISSN: 0386-2178  
DOCUMENT TYPE: Journal; General Review  
LANGUAGE: Japanese

AB A review with 16 refs. is given on the techniques of multidimensional gas chromatog. (MDGC). The applications of MDGC to the sepn. of traces of xylene from ethylbenzene, optical resolution of the iso-Pr urethane derivs. of 4 menthol isomers, and the determination of very small amts. of 2-butanol, ProH, 2-methylpropanol, BuOH, 2-methylbutanol, and pentanol in H<sub>2</sub>O are discussed.

L7 ANSWER 3 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1979:512892 CAPLUS  
DOCUMENT NUMBER: 91:112892  
TITLE: Sorption of trace impurities. Dependence of coefficients of trace impurity-principal substance separation on the degree of sorbent saturation and on temperature  
AUTHOR(S): Tolmachev, A. M.; Egorov, E. N.; Trubnikov, I. B.  
CORPORATE SOURCE: Mosk. Univ., Moscow, USSR  
SOURCE: Zhurnal Fizicheskoi Khimii (1979), 53(6), 1550-3  
CODEN: ZFKHA9; ISSN: 0044-4537  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian

AB By using the exptl. sorption data for mixts. of C<sub>6</sub>H<sub>6</sub>, 1-hexene, octane, and hexane on zeolite NaX and C BAU, it was shown that the separation coeffs. of impurity-basic compound depend on relative concentration of the basic compound in liquid and sorbent and agrees well with the previously developed stoichiometric theory of sorption. The dependence was studied of equilibrium coefficient K and separation coefficient ( $\alpha$ ) on temperature and thermodyn. parameters of exchange adsorption as function of C-atom number in homologous microimpurity mols. A significant contribution of entropy to the  $\alpha$  and K values was observed

L7 ANSWER 4 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1970:441613 CAPLUS  
DOCUMENT NUMBER: 73:41613  
TITLE: Chromatographic study of products from the oxidative ammonolysis of xylenes  
AUTHOR(S): Aliev, M. I.; Mekhtiev, S. D.; Kyazimov, E. A.; Samedov, Kh. S.  
CORPORATE SOURCE: USSR  
SOURCE: Azerbaidzhanskii Khimicheskii Zhurnal (1969), (5), 52-5  
CODEN: AZKZAU; ISSN: 0005-2531  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian



AB The end products were iso- and terephthalonitriles, but the reaction product contained traces of xylenes and tolunitriles. NaCl crystals and N at 150° were used as carriers for the anal. of the solid reaction product. The impure terephthalonitrile contained 35.5% isophthalonitrile, 3.4% m-tolunitrile (I), and traces of MeCN and xylene. The liquid product consisted of 82.5 I and 16.9% m-xylene. The chromatog. anal. took .apprx.10 min.

L7 ANSWER 5 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1967:464235 CAPLUS  
DOCUMENT NUMBER: 67:64235  
TITLE: Preparation of 4-(3-lindolyl)hexahydro-1H-azepines  
PATENT ASSIGNEE(S): Upjohn Co.  
SOURCE: Neth. Appl., 12 pp.  
CODEN: NAXXAN  
DOCUMENT TYPE: Patent  
LANGUAGE: Dutch  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
NL 6608568		19661223	NL 1966-8568	19660621
DE 1620634			DE	
FR 1484226			FR	
GB 1155193			GB	
GB 1155194			GB	
GB 1155195			GB	
US 3347866		19671017	US 1965-466102	19650622
US 3396160		19680806	US 1966-588713	19661024
PRIORITY APPLN. INFO.:			US	19650622

GI For diagram(s), see printed CA Issue.

AB The quaternary salts of the title compds. (I) with fluosilicic acid are moth-proofing agents; I show anti-tumor, antiviral, antibacterial, and stimulating activity; The intermediates (II) show antitremor and strychnineantagonist activity. I are prepared by the reduction of II with LiAlH<sub>4</sub>; II are obtained by the decarboxylation of III (R<sub>2</sub> = H) by heating the acid at its m.p. under reduced pressure. III (R<sub>2</sub> = H) are obtained from the corresponding esters by refluxing them in a basic aqueous solution;

the

esters are obtained by the reaction of IV with a malonate, in the presence of a strong base in an inert solvent. Thus, a mixture of 50 g. IV, 42 g. CH<sub>2</sub>(CO<sub>2</sub>Et)<sub>2</sub>, and 1 g. powdered NaOH in 300 ml. xylene is refluxed 31.25 hrs. in N, 500 mg. powdered NaOH is added after 7.5 and 25.5 hrs. The EtOH is separated and the cooled reaction mixture is poured into dilute AcOH and extracted

with CHCl<sub>3</sub>. The extract is dried, concentrated in vacuo; the last traces of xylene are eliminated by azeotropic distillation and the residue taken up in C<sub>6</sub>H<sub>6</sub>. The solution is chromatographed on 900 g. neutral Al<sub>2</sub>O<sub>5</sub> and eluted with 50% Et<sub>2</sub>O-CHCl<sub>3</sub> to give 11.1 g. III (R<sub>2</sub> = Et, isomer A) (V), m. 196.5-8.0° (MeOH-EtOAc). Concentration of the mother liquors from the crystallization gives a brown oil, which is dissolved in 150 ml. anhydrous MeOH.

The ice-cold solution is treated with 30 ml. 0.976N aqueous NaOH, and the solution

is heated to 25°. After 24 hrs., H<sub>2</sub>O is added and the MeOH eliminated to give III (R<sub>2</sub> = Et, isomer B), m. 212.5-14.5° (CHCl<sub>2</sub>-MeOH), mixed m.p. of A and B isomers: 201.0-12.5°. A solution of 1 g. V in 50 ml. anhydrous EtOH is treated with 7.33 ml. of a 0.433N aqueous KOH solution and refluxed gently 7 hrs. in a N atmospheric. The solution is concentrated in

vacuo, the residue suspended in H<sub>2</sub>O, and the aqueous extract cooled on ice and acidified with concentrated HCl to give 82.9% III (R<sub>2</sub> = H), m. 151.5-4.0° (decomposition), of which 0.598 g. is heated 5 min. in a small vessel at 178°/14 mm. The obtained colorless glass gives 89.5% II, m.

148-50° (EtOAc). II (6.0 g.) is added under N with stirring to an ice-cold solution of 6.0 g. LiAlH<sub>4</sub> in 600 ml. tetrahydrofuran. The mixture is refluxed 6.66 hrs. with stirring and left 18 hrs. at 25°, then cooled to 0° and treated successively with 6 ml. H<sub>2</sub>O, 0.6 ml. aqueous NaOH, and 6 ml. H<sub>2</sub>O. The inorg. precipitate is separated and the filtrate concentrated to give 89.8% I, m. 81-5° (Et<sub>2</sub>O-Skellysolve B).

L7 ANSWER 6 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1965:43116 CAPLUS  
DOCUMENT NUMBER: 62:43116  
ORIGINAL REFERENCE NO.: 62:7566c-e  
TITLE: Catalysts for isomerization of alkyl aromatic compounds  
INVENTOR(S): Pfefferle, William C.  
PATENT ASSIGNEE(S): Engelhard Industries, Inc.  
SOURCE: 19 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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FR 1375730		19641023	FR 1963-934898	19630515
GB 993223			GB	
PRIORITY APPLN. INFO.:			US	19620518

AB An isomerization-cracking catalyst of 1.19-2.38 mm. particle size was prepared from a mixture of 3.72 kg. of SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub> powder containing 0.5% Mn and 5.6 kg. Al hydrate powder containing 0.8% Pt. In a reactor, 15 g. of the catalyst was swept with N, then reduced 2 hrs. with H at 482°. A charge of 96.8% PhEt containing traces of xylenes, 0.6 ppm. S, and 0.1 ppm. organic N, was recycled for 6-14 hrs. at 454°, 15.95 sec. contact time, and 12.3 atmospheric to give a product consisting of C<sub>6</sub>H<sub>6</sub> 5.1, PhMe 3.4, o-xylene 9.0, m-xylene 19.2, p-xylene 10.8, and ethylbenzene 45%. Passing the effluent gas through a Cu chloride solution to eliminate NH<sub>3</sub> before recycling decreased the required contact time from 15 to 12 sec. H containing 2.5 ppm. N, when used to recycle a mixture of o-xylene 18, m-xylene 48, p-xylene 10, PhEt 24%, and 0.7 ppm. S gave after 6-13 hrs. at 453.9°, 15.91 sec. contact time, and 12.6 atmospheric, a product containing C<sub>6</sub>H<sub>6</sub> 0.0, PhMe 1.0, o-xylene 18.6, m-xylene 44.2, p-xylene 12.0, and PhEt 21.6%.

L7 ANSWER 7 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:401736 CAPLUS  
DOCUMENT NUMBER: 59:1736  
ORIGINAL REFERENCE NO.: 59:240d-f  
TITLE: Recovery of palladium from catalysts  
INVENTOR(S): Lait, Raymond; Lloyd-Owen, Dennis R.  
PATENT ASSIGNEE(S): Laporte Chemicals, Ltd.  
SOURCE: 6 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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GB 922021		19630327	GB 1959-2432	19590122
PRIORITY APPLN. INFO.:			GB	19590122

AB Spent, Al<sub>2</sub>O<sub>3</sub>-supported Pd catalyst, such as that used in the manufacture of H<sub>2</sub>O<sub>2</sub>, is washed with xylene to remove organic matter, steamed at 90° to remove the last traces of xylene, and then stirred in a hot alkaline solution of HCHO to ensure complete reduction of the Pd. The reduced catalyst is stirred with 8N HCl and Cl<sub>2</sub> is passed through the mixture

to dissolve the Pd; the mixture is filtered and the Al<sub>2</sub>O<sub>3</sub> is washed with aqueous NaOH solution, then with water, and dried at 120° for reuse. Pd is precipitated from the filtrate by immersing a sheet of Al in the solution, and the Pd is filtered off and washed with water and dilute NaOH solution. The Pd can be dissolved in HCl and Cl and used in the preparation of fresh catalyst.

L7 ANSWER 8 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1962:7447 CAPLUS  
DOCUMENT NUMBER: 56:7447  
ORIGINAL REFERENCE NO.: 56:1356e-i,1357a  
TITLE: Dicarbamates  
PATENT ASSIGNEE(S): Carter Products, Inc.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 873243		19610719	GB 1960-9068	19600315

AB Dicarbamates were prepared (a) by reaction of the appropriate 2,2-dialkyl-1,3-propanediol (I) with COCl<sub>2</sub> in the presence of NaOH to form the corresponding di(chlorocarbonate), which was converted to the dicarbamate by reaction with either aqueous or anhydrous NH<sub>3</sub>. (b) from I by ester exchange, using low mol. weight urethans, and (c) by reaction of I with cyanic acid in anhydrous medium. I was prepared by any known method, e.g., by reduction of the corresponding disubstituted malonic acid. Thus, 2-methyl-2-(1-ethylpropyl)-1,3-propanediol dicarbamate (II) was prepared as follows: 16.5 g. NaH was added to a stirred solution of 112 g. diethyl methylmalonate in 1000 ml. anhy. PhMe; after H evolution was complete, 116 g. 3-bromopentane in 500 ml. C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> was added, the solution refluxed 36 hrs., NaBr filtered off, the filtrate distilled (to remove PhMe), and the residue distilled to give 56 g. diethyl (methyl-1-ethylpropyl)malonate (III), b<sub>9</sub> 116-27°, n<sub>25D</sub> 1.4377, 55 g. of which was reduced with 13 g. LiAlH<sub>4</sub> to give 2-methyl-2-(1-ethylpropyl)-1,3-propanediol (IV), b<sub>0</sub> 87-92°, n<sub>25D</sub> 1.4621, m. 45-8° (petr. ether). (Iso-Pro)<sub>3</sub>Al (1.6 g.) was added to 16 g. IV and 19.6 g. ethyl urethan in 300 ml. anhydrous xylene and the mixture distilled to remove EtOH formed; xylene was distilled under reduced pressure, 200 ml. H<sub>2</sub>O added, and the mixture distilled to remove final traces of xylene. The residue was dissolved in 200 ml. hot 20% iso-ProH in H<sub>2</sub>O and the solution filtered and cooled to give II, m. 118-20°, in 40% yield, approx. 0.1% soluble in H<sub>2</sub>O. II was also prepared from IV by methods (a) and (c). The 2-Et analog (V) of II was prepared as follows: 1 mole diethyl ethylmalonate in 1500 ml. C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> was treated with 1.1 moles 3-bromopentane (as described for III) to give 95 g. diethyl (ethyl-1-ethylpropyl)malonate (VI), b<sub>10</sub> 131-8°, n<sub>25D</sub> 1.4367. VI (90 g.) on reduction with 20.1 g. LiAlH<sub>4</sub> gave 41.5 g. 2-ethyl-2-(1-ethylpropyl)-1,3-propanediol (VII), b<sub>0</sub> 76-7°, n<sub>25D</sub> 1.4643. V (8.6 g.), m. 119-21°, was prepared from 17.4 g. VII (solubility of V in H<sub>2</sub>O was approx. 0.1%). These dicarbamates, more particularly II and V, had highly effective sleep producing characteristics. When heated or boiled with acid or alkali, they hydrolyzed to the corresponding 2,2-disubstituted 1,3-propanediol, NH<sub>3</sub>, and CO<sub>2</sub>.

L7 ANSWER 9 OF 12 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1961:22704 CAPLUS  
DOCUMENT NUMBER: 55:22704  
ORIGINAL REFERENCE NO.: 55:4468e-i,4469a-c  
TITLE: Intermediates necessary in the synthesis of resinols and their derivatives. V  
AUTHOR(S): Traverso, Giorgio

CORPORATE SOURCE: Univ. Pavia, Italy  
SOURCE: Gazzetta Chimica Italiana (1959), 89, 1810-17  
CODEN: GCITA9; ISSN: 0016-5603  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB cf. CA 53, 20025i; 54, 2282g. In an attempt to prepare 3,4,2,5-Me<sub>2</sub>[3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub>C<sub>4</sub>O (I), required for investigation of the natural resinols olivil and isoolivil (Haworth and Atkinson, CA 32, 66571) the procedure according to Haworth, et al. (CA 53, 21825d) gave the corresponding alc., 3,4,2,5-Me(OHCH<sub>2</sub>) [3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub>C<sub>4</sub>O (II). RCOCH<sub>2</sub>CO<sub>2</sub>Et [R = 3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>] (3 g.) in 25 ml. warm alc. and 0.35 g. Na in 8 ml. absolute alc. treated with 3 ml. dry MeI and the mixture refluxed 3 hrs., the cooled mixture poured into H<sub>2</sub>O and extracted with Et<sub>2</sub>O or C<sub>6</sub>H<sub>6</sub>, the dried extract evaporated,

and the residue distilled gave RCOCHMeCO<sub>2</sub>Et, b<sub>3</sub> 202-5°. The ester (5 g.) in 25-30 ml. alc. boiled with 2 g. NaOH in 25 ml. H<sub>2</sub>O and the mixture refluxed 1.5 hrs., diluted with excess H<sub>2</sub>O and kept 7-8 hrs. at 0°, the washed and dried crystalline product (3-3.5 g., m. 56-60°) recrystd. from petr. ether (b. 50-70°), or distilled at 1-2 mm. gave RCOEt, m. 62°. The ketone (3.5 g.) in 40 ml. CHCl<sub>3</sub> stirred with a few drops of 48% HBr and kept 15 min., treated with 2-3 ml. Br in CHCl<sub>3</sub> (2.5 g./10 ml.) in direct sunlight at 50-60° to permanent coloration and the remainder added portionwise at 20°, the mixture stirred 20-30 min. and washed with H<sub>2</sub>O and 5% aqueous Na<sub>2</sub>CO<sub>3</sub>, the dried CHCl<sub>3</sub> extract evaporated

in

vacuo below 40°, and the residue crystallized from petr. ether gave 4 g. 3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>COCHBrMe (III), m. 80°. III (4 g.) in 50 ml. xylene refluxed 24 hrs. with 4 g. activated Cu and the filtered solution evaporated in vacuo on a steam bath, the last traces of xylene removed by steam distillation and the residue refluxed 1 hr. with 40 ml. MeOH saturated with dry HCl, the brown solution diluted with H<sub>2</sub>O and

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hrs. at 0°, the residual paste taken up in a min. of hot MeOH and refrigerated 12 hrs. gave II, C<sub>22</sub>H<sub>28</sub>O<sub>6</sub>, m. 152°, also produced by substituting electrolytic Ag for the activated Cu. Na(0.2 g.) in 20 ml. alc. added at 50° to 2 g. 3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>COCH<sub>2</sub>CO<sub>2</sub>Et in 10 ml. alc. and immediately after initiation of the reaction the mixture treated with 2.2 g. III in 10 ml. alc. at 50°, the mixture kept 3 days at room temperature and refluxed 30 min., concentrated to 15 ml. in vacuo and diluted

with Et<sub>2</sub>O,

stirred with H<sub>2</sub>O, and the dried organic layer evaporated in vacuo gave an oily stereoisomeric mixture of esters, RCOCHMeCH(CO<sub>2</sub>Et)COR (IV). IV (2 g.) refluxed in 20-30 ml. MeOH saturated with dry HCl and the dark brown solution diluted with H<sub>2</sub>O, refrigerated 12 hrs., and the residue on decantation taken up in MeOH gave 1.2 g. 3,4,2,5-Me(EtO<sub>2</sub>C) [3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub>C<sub>4</sub>O (V), m. 116-17°. V (0.5 g.) in 30 ml. dry C<sub>6</sub>H<sub>6</sub> kept 12 hrs. with 0.15 g. LiAlH<sub>4</sub> in 25 ml. dry Et<sub>2</sub>O and the mixture decomposed with ice and a slight excess of dilute HCl, the aqueous layer washed with C<sub>6</sub>H<sub>6</sub> and the combined dried (Na<sub>2</sub>SO<sub>4</sub>) C<sub>6</sub>H<sub>6</sub> layers evaporated in vacuo, the residue taken up in hot MeOH, and the solution chilled gave 0.35-0.40 g. II; Ac derivative m. 136°. V (0.5 g.) in 15 ml. 95% alc. refluxed 30 min. with 0.6-0.8 g. KOH and the mixture refluxed 30 min. with 5 ml. H<sub>2</sub>O, diluted with excess H<sub>2</sub>O, and acidified with dilute HCl gave 0.40 g. product, recrystd. from EtOH or MeOH to give 3,4,2,5-Me(HO<sub>2</sub>C) [3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub>C<sub>4</sub>O (VI), m. 162°. VI (0.2 g.) and 0.1 g. bronze dust homogenized in a mortar and decarboxylated at 230-50°/1.0 mm. and the product distilled, the distillate taken up in hot MeOH, and the cooled mixture filtered gave 3,2,5-Me [3,4-MeO(EtO)C<sub>6</sub>H<sub>3</sub>]<sub>2</sub>C<sub>4</sub>HO (VII), m. 116°. IV (1.5 g.) in 15 ml. alc. added dropwise to 0.7 g. NaOH in 15 ml. boiling H<sub>2</sub>O and the solution refluxed 1 hr., diluted with H<sub>2</sub>O, and extracted with C<sub>6</sub>H<sub>6</sub> gave an almost quant. yield of the oily diketone, RCOCHMeCH<sub>2</sub>COR, C<sub>23</sub>H<sub>28</sub>O<sub>6</sub>, distilling at 1 mm. The diketone (0.5 g.) kept 12 hrs. at 0° and the residue on decantation crystallized from MeOH gave 0.3 g. VII.

ACCESSION NUMBER: 1960:56136 CAPLUS  
DOCUMENT NUMBER: 54:56136  
ORIGINAL REFERENCE NO.: 54:10904f-i,10905a-c  
TITLE: Chemistry of xylylenes. IV. Stabilization of benzyl radicals in solution  
AUTHOR(S): Errede, L. A.; Cassidy, J. P.  
CORPORATE SOURCE: Minnesota Mining and Mfg. Co., St. Paul  
SOURCE: Journal of Organic Chemistry (1959), 24, 1890-2  
CODEN: JOCEAH; ISSN: 0022-3263  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB cf. C.A. 52, 8986e. Solns. of benzyl radicals, in concns. as high as  $10^{-4}$  molar, were prepared by fast flow pyrolysis of PhMe at low pressure and subsequent condensation of the pyrolyzate into a solvent kept at  $-78^{\circ}$ . Solvated radicals were still present 1.5 h. after termination of pyrolysis. Surprisingly, an equal amount of p-xylylene (I) was also accumulated during the reaction. Apparently I was formed by dehydrogenation of p-xylene produced via methylation of PhMe. The unusual stability of solvated benzyl radicals at  $-78^{\circ}$  may be a manifestation of complex formation with PhMe thereby decreasing their rate of coupling. Gas chromatog. and IR anal. indicated that the composition of the PhMe used was 99.8% pure, 0.1% C<sub>6</sub>H<sub>6</sub>, and 0.1% aliphatic hydrocarbons. PhMe (500 g.) was subjected to gas flow pyrolysis at  $970^{\circ}/5$  mm. and 0.02 s. residence time and the pyrolyzate condensed at  $-78^{\circ}$ ; the 480 g. volatile components were separated by distillation at atmospheric pressure.

The composition was determined by gas chromatog. and IR anal. to be 98.9% PhMe, 0.7% C<sub>6</sub>H<sub>6</sub>, 0.4% PhEt, 0.1% aliphatic hydrocarbons, and a trace of xylenes. The 2.3 g. residue, b. above  $140^{\circ}$ , was separated by distillation at 1 mm. The 1st fraction, 1.9 g., b.  $85-100^{\circ}$ , was recrystd. from MeOH at  $-78^{\circ}$  to afford 1.5 g. crude Ph<sub>2</sub>, m.  $49-50^{\circ}$ . The mother liquors were evaporated to dryness to leave 0.4 g. oil as residue, identified as a mixture of Ph<sub>2</sub>CH<sub>2</sub> and 2-methyldiphenylmethane with a small amount of stilbene. The 2nd fraction, 0.25 g., was identified as impure anthracene, platelets, m.  $210-12^{\circ}$ . Thus, 5.4 mol PhMe was metered into the system and 4.1 mol recovered. The rest was converted into 0.04 mol C<sub>6</sub>H<sub>6</sub>, 0.02 mol PhEt, 0.007 mol Ph<sub>2</sub>, 0.002 mol Ph<sub>2</sub>CH<sub>2</sub>, 0.001 mol anthracene, and traces of xylenes and stilbene. The materials balance accounted for 96% of the Ph groups metered into the furnace. PhMe (500 g.) was vaporized at atmospheric pressure and the gas metered at the rate of

0.03 mol/min. to the evacuated pyrolysis system, the gas pyrolyzed at  $1000^{\circ}/4$  mm. and 0.004 s. residence time, and the pyrolyzate condensed at  $-78^{\circ}$  in 4.5 l. PhMe. A 2nd 4 l. solution of pyrolyzate in hexane was prepared as above via fast flow pyrolysis of 500 g. PhMe; the solution contained about  $10^{-3}$  mole of titratable species when quenched at  $-78^{\circ}$  with excess iodine. The resulting solution was warmed to room temperature, the excess iodine reduced with aqueous Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and the organic solvent

removed by evaporation to give 11 g. dark residual oil. This was treated at  $50^{\circ}$  with 10 g. NaCN in 200 cc. 1:1 H<sub>2</sub>O-MeOH during 4 h., the mixture separated, extracted with Et<sub>2</sub>O, evaporated, the residue leached 4 h. with aqueous NaOH and

the solution extracted with Et<sub>2</sub>O; IR anal. indicated that the product consisted essentially of the usual products of fast flow pyrolysis of PhMe. Evaporation of the Et<sub>2</sub>O extract gave 1.1 g. acidic material as a residue, this was separated

by vacuum sublimation. The more volatile acid fraction of 0.5 g. was recrystd. from hexane to give phenylacetic acid, m.  $74.5-6.0^{\circ}$ . The 0.3 g. of less volatile acid gave p-phenylenediacetic acid, m.  $243-5^{\circ}$ . The above results indicated that the benzyl radical and I were present in the original solution in at least the amts. of the corresponding acids isolated.

ACCESSION NUMBER: 1921:6922 CAPLUS  
DOCUMENT NUMBER: 15:6922  
ORIGINAL REFERENCE NO.: 15:1236g-h  
TITLE: Phenolphthalein  
AUTHOR(S): Utz, F.  
CORPORATE SOURCE: Suddeut  
SOURCE: Apoth. Ztg. (1920), 60, 430-1,442-4  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The amts. of phenolphthalein in g. contained in 100 cc. of a warm saturated solution of the substance in the various solvents are as follows: EtOH, 20.91; MeOH, 14.18; acetone, 26.39; CHCl<sub>3</sub>, 3.06; CS<sub>2</sub>, 0.18; ether, 5.92; light petroleum, 0.18; or 1.06, depending on the b. p. of the solvent; benzene, 0.16; CCl<sub>4</sub>, traces; xylene, 0.18; toluene, 0.61; nitrobenzene, 4.41; ethyl acetate, 6.57. Water (10 cc.) dissolves 0.0002 g. of phenolphthalein at the ordinary temperature, and about ten times this amount when warmed.

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ACCESSION NUMBER: 1909:2301 CAPLUS  
DOCUMENT NUMBER: 3:2301  
ORIGINAL REFERENCE NO.: 3:461a-c  
TITLE: The Analysis of Sausage  
AUTHOR(S): Kreis, H.  
CORPORATE SOURCE: Basle  
SOURCE: Chemiker-Zeitung (1909), 32, 1042-5  
CODEN: CMKZAT; ISSN: 0009-2894  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The determinations described are water, fat, and water and fat-free residue. Water is determined by introducing 50 g. of the well mixed sample into a short-necked 200 cc. Kjeldahl flask, adding 100 cc. of xylene and distilling until practically dry. The mixture of xylene and water is allowed to stand until the two layers separate (generally over night) and the water is measured in a burette. Other substances which are immiscible with water and boil above 100° were tried but xylene is preferred. Fat is determined in the residue by refluxing with benzene which is then drawn off, made to 200 cc. with more benzene and 20 cc. of this solution dried in a tared dish, in a current of air, on a glycerol bath heated to 150° to remove last traces of xylene. The substance remaining in the flask is dried in the same manner and weighed as the water and fat-free residue. The calculated water content of the fat-free solids is pointed out as a valuable factor in determining the quality of the sausage. Some tables are given showing the water, fat and residue in sausage at successive stages of its manufacture from the raw meat to the finished product.

=> S TOLUENE AND PURITY AND TRACES AND XYLEN?

172318 TOLUENE  
1526 TOLUENES  
172885 TOLUENE  
(TOLUENE OR TOLUENES)  
173115 PURITY  
4323 PURITIES  
176107 PURITY  
(PURITY OR PURITIES)  
75330 TRACES  
126756 XYLEN?

L8 8 TOLUENE AND PURITY AND TRACES AND XYLEN?

=> D 1-8 IBIB ABS

L8 ANSWER 1 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN  
ACCESSION NUMBER: 1969:464518 CAPLUS

DOCUMENT NUMBER: 71:64518  
TITLE: Temperature dependence of the surface tension of gallium and its interfacial tension at the interface with some nonpolar organic liquids  
AUTHOR(S): Karashaev, A. A.; Zadumkin, S. N.; Kukhno, A. I.  
CORPORATE SOURCE: USSR  
SOURCE: Poverkh. Yavleniya Rasplavakh (1968), 219-25.  
Editor(s): Eremenko, V. N. "Naukova Dumka": Kiev, USSR.  
CODEN: 21HMA8  
DOCUMENT TYPE: Conference  
LANGUAGE: Russian

AB The nonlinear nature of the temperature dependence of the surface tension  $\sigma$  of Ga is caused by the insufficiently high purity of the metal investigated. Some of the previous works did not specify the degree of purity of the Ga being used. The calculated  $\sigma$  and  $d\sigma/dT$  values are significantly higher than the resp. exptl. values. This work was undertaken to explain these discrepancies. The Ga used was 99.9998% pure, and contained traces of Al, Cu, and In. Inasmuch as Ga can easily be supercooled, its  $\sigma$  at temperature significantly lower than its m.p. was also determined. The large drop method was used to measure the  $\sigma$  of Ga. Independent measurements (14) of  $\sigma$  gave very reproducible results. The polytherm of the surface tension of Ga in the temperature range 20-360° can be given by the equation:  $\sigma = 718 - 0.101(t - 29.87)$ . Expts. performed with 99.9% Ga gave essentially the results reported by previous works. It is doubly confirmed that the surface tension of supercooled Ga of 99.9998% purity at 20° is 719 mj./m.2. In a number of cases the expts. were performed not only in liquid, but also in saturated gases. The interfacial tension of Ga at the boundary with the following organic liquids at 20° was determined: benzene, toluene, p-xylene, hexane, octane, decane, CCl<sub>4</sub>, Me<sub>2</sub>CO, heptane, nonane, pentane, dodecane. The values are tabulated. The saturated vapors of organic liqs. significantly lower the surface tension of Ga.

L8 ANSWER 2 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1960:38963 CAPLUS  
DOCUMENT NUMBER: 54:38963  
ORIGINAL REFERENCE NO.: 54:7639g-i,7640a-i,7641a  
TITLE: Synthesis of some monomers on alumino-chromium catalysts  
AUTHOR(S): Kotlyarevskii, I. L.; Fisher, L. B.; Zanina, A. S.; Terpugova, M. P.; Volkov, A. N.; Shvartsberg, M. S.  
SOURCE: Izvestiya Vysshikh Uchebnykh Zavedenii, Khimiya i Khimicheskaya Tekhnologiya (1959), 2, 608-13  
CODEN: IVUKAR; ISSN: 0579-2991  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The optimal conditions of the alkylation of iso-C<sub>4</sub>H<sub>10</sub> by C<sub>2</sub>H<sub>4</sub> in the presence of AlCl<sub>3</sub> were established: 48-51°, 7-9 atmospheric, 1.5 kg. iso-C<sub>4</sub>H<sub>10</sub>, 145 g. C<sub>2</sub>H<sub>4</sub>, 40 g. EtCl, 50 g. AlCl<sub>3</sub>, 2 hrs. Under these conditions, the yield in alkylate reached 300% with respect to C<sub>2</sub>H<sub>4</sub> and 30% with respect to iso-C<sub>4</sub>H<sub>10</sub>, and the content of 2,3-dimethylbutane (I) reached 45-7%. A decrease in temperature from 50 to 35° decreased by 3 times the yield in alkylate and an increase of 10-15° above the optimum temperature decreased by 3 times the yield of I, but that of the alkylate remained constant. A high content of I was observed only when the addition of C<sub>2</sub>H<sub>4</sub> was continued during the whole process, and the partial pressure of C<sub>2</sub>H<sub>4</sub> did not exceed 0.5-0.7 atmospheric at a total pressure of 7-9 atmospheric. The high-temperature alkylation of iso-C<sub>4</sub>H<sub>10</sub> by C<sub>2</sub>H<sub>4</sub> was carried out under the following optimal conditions: 450°, 200 atmospheric, 25 min., 10:1 iso-C<sub>4</sub>H<sub>10</sub>-C<sub>2</sub>H<sub>4</sub> (by weight), 2.5% of initiator; the yield of alkylate reached 170-80% (on C<sub>2</sub>H<sub>4</sub>) and the main product was 2,2-dimethylbutane(II).

2,3-Dimethyl-1,3-butadiene(IIa) was prepared from I in 2 steps: (1) dehydrogenation of I to tetramethylethylene and 2,3-dimethyl-1-butene(IIb), (2) dehydrogenation of the olefins formed to the corresponding diene. The dehydrogenation of I was studied on an Al-Cr catalyst in which 50% Al<sub>2</sub>O<sub>3</sub> was replaced by "Troshkov kaolin" (KIT) and on the industrial catalyst K-5. The best reproducibility of KIT was attained at 550° and a volume rate of 2-2.5 hrs.<sup>-1</sup>; the catalytic products, under these conditions, amounted to 75% and contained about 60% unsatd. and 12% dienic hydrocarbons (14-15% at 570°), that consisted of 90% IIa and 10% isoprene. The activity of K-5 was lower than that of KIT, but its use resulted in less cracking; the optimal conditions with K-5 were 550° and 2 hrs.<sup>-1</sup>, and the yield reached 83.5% (49% of unsatd. hydrocarbons). A mixture of I and IIb on K-5 in the presence of dilute H (1:4) at 630° and a volume rate of 2 hrs.<sup>-1</sup> yielded 78.7% of unsatd. hydrocarbons, in which IIa amounted to 26%. The dehydrogenation of II attained a lower degree than that of I, owing to the presence of a quaternary C atom, and was accompanied by much cracking: under optimal conditions on K-5 (570, 2 hrs.<sup>-1</sup>), the catalytic products were 31.4% unsatd. hydrocarbons, in which 3,3-dimethyl-1-butene and tetramethylethylene amounted to 48.4 and 17.7%, resp., and the rest consisted of pentenes and isoprene. The aromatization of divinyl-acetylene(III) hydrocarbons yielded a series of mono-, di-, and tricyclic hydrocarbons: the aromatization of III at 500° during 11.5 sec. in a stream of H over 20% Cr-Al catalyst yielded 34% of catalytic products, half of which was C<sub>6</sub>H<sub>6</sub>. By the same method, vinylisopropenylacetylene on 20:80 Cr<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> or 2:18:80 MgO-Cr<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> yielded 28.2% (400°, 0.2 kg./l. catalyst hr.) and 11% toluene, resp. The aromatization of diisopropenylacetylene at 400° in a stream of H and a volume rate of 0.2 kg./l. catalyst hr. yielded 28% p-xylene; in the same manner 2,5-dimethylhepta-1,5-dien-3-yne yielded 1,2,4-trimethylbenzene, and 3,6-dimethylocta-2,6-dien-4-yne yielded 1,2,3,4-tetramethylbenzene. The aromatization of isopropenylcyclohexenylacetylene yielded β-methylnaphthalene and dicyclohexenylacetylene, phenanthrene. Thus III hydrocarbons, easily obtained from acetylene alcs. and glycols, served as initial substances in the preparation of aromatic hydrocarbons of various classes, with the structure 1,2,3,4-R<sub>3</sub>R<sub>2</sub>R<sub>1</sub>RC<sub>6</sub>H<sub>2</sub>. In most cases, best results were observed when III hydrocarbons were hydrogenated by 2 moles of H before aromatization. A method was developed for the industrial preparation of p-xylene: the 1st step, the condensation of C<sub>2</sub>H<sub>2</sub> with Me<sub>2</sub>CO, was carried out by the Favorskii reaction under the influence of powdered KOH (which could be recovered); a yield of 85-90% was obtained in various solvents; the 2nd step, the hydrogenation of acetylene γ-glycol to saturation took place in a nearly quant. yield, over skeletal Ni at 80-100° and a H pressure of 10-20 atmospheric; the 3rd step, the cyclization of saturated glycol into 2,2,5,5-tetramethyltetrahydrofuran took place in a 85-90% yield, by distilling the glycol with traces of H<sub>3</sub>PO<sub>4</sub> in slight vacuum; at least the aromatization of tetrahydrofuran into p-xylene took place in a 60-5% yield, on 2:18:80 MgO-Cr<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> in a slow stream of H at 500-10° and a volume rate of from 0.3 to 0.4 kg./l. catalyst hr.; the product obtained was of high purity and did not contain o- or m-xylenes. 1,4,4-Trimethyl-1-phenylbutynediol and ethylphenylvinylethynylcarbinol were aromatized on 2:18:80 MgO-Cr<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> at 430° and a volume rate of 0.3 g. kg./l. catalyst hr. into p-methylbiphenyl in an 11% to 3% yield. To increase the yield (to 50%), acetyleneglycol and vinylacetylene alc. were partially and completely hydrogenated under optimal conditions (555°, 0.66 kg./l. catalyst hr.). 2,2,5-Trimethyl-5-phenyltetrahydrofuran easily obtained by the dehydration of 1,1,4-trimethyl-4-phenylbutane-1,4-diol, behaved the same way. The above transformations showed a simple way to the synthesis of polyphenyl systems; these could be used as scintillators and, apparently, as monomers of the terephthalic acid or p-di(aminomethyl)benzene type of the general formula: RC<sub>6</sub>H<sub>4</sub>(C<sub>6</sub>H<sub>4</sub>)<sub>n</sub>C<sub>6</sub>H<sub>4</sub>R, where R is CO<sub>2</sub>H or CH<sub>2</sub>NH<sub>2</sub> and n = 0, 1, 2, 3, etc. It is supposed that the polycondensation of such monomers with dicarboxylic acids, glycols, glycerols, and diamines would



lead to fibers having interesting properties. As a 1st example, p,p'-dimethylbiphenyl was prepared

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ACCESSION NUMBER: 1960:16947 CAPLUS

DOCUMENT NUMBER: 54:16947

ORIGINAL REFERENCE NO.: 54:3392h-i,3393a-i,3394a-c

TITLE: Synthesis of p-nitrophenyl substituted tetrazolium salts containing iodine and other groups

AUTHOR(S): Karmarkar, Shankar S.; Barnett, Russell J.; Nachlas, Marvin M.; Seligman, Arnold M.

CORPORATE SOURCE: Johns Hopkins Univ., Baltimore, MD

SOURCE: Journal of the American Chemical Society (1959), 81, 3771-5

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. C.A. 52, 8256c. The syntheses of several tetrazolium salts were described. These included an improved method for preparing the ditetrazolium salt (Nitro-BT) (I) and 2-iodo-5-phenyl-substituted analogs of I for possible use in electron microscopy, and 5 analogs of 2-(p-nitrophenyl)-substituted monotetrazolium salts containing 3-Ph substituents of varying electronegativity for a study of their ability to act as electron acceptors with various dehydrogenases. Powdered [3,4-MeO(H<sub>2</sub>N)C<sub>6</sub>H<sub>3</sub>]22H Cl (Ia) (50 g.) in 240 ml. H<sub>2</sub>O treated with 60 ml. HCl, the solution cooled to 0°, diazotized with 24 g. NaNO<sub>2</sub> in 60 ml. H<sub>2</sub>O, the product added to 77 g. p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHNCHPh in 700 ml. tetrahydrofuran, treated with 40 g. KOH in 75 ml. H<sub>2</sub>O (temperature kept at -20 to -25°), allowed to come to room temperature after about 1 hr., diluted with 320 ml. H<sub>2</sub>O and 700 ml. MeOH, filtered, and the precipitate washed with MeOH followed by 2400 ml. boiling H<sub>2</sub>O yielded a crude mixture of mono- (II) and diformazan (III). The precipitate

extracted

(Soxhlet) 3 days in 3 portions with dioxane or C<sub>6</sub>H<sub>6</sub>, the residue dried, powdered, and reextd. (the cycle repeated 5 times after extracting 2 days) yielded

(total) 70 g. III, m. 255° (decomposition), containing traces of II. III (5 g.) in 80 ml. tetrahydrofuran and 80 ml. dioxane treated with 6 ml. iso-AmNO<sub>2</sub>, the mixture saturated with dry HCl at 0° (2 ml. iso-AmNO<sub>2</sub> added during the 2 hrs.), the mixture stirred 4 hrs. at room temperature, filtered with C, the solvents removed in vacuo, and the residue in EtOH added to dry Et<sub>2</sub>O yielded 4.5 g. I, m. 184° (decomposition). The slightly impure I thus obtained reduced to III with glucose-NH<sub>4</sub>OH, the product extracted with EtOH and xylene (Soxhlet), and the III, m. 270-2°, reoxidized yielded I which was still slightly impure.

Several other procedures were tried for purification but none yielded a histochem. pure product. A test was described for determining the

purity of I. p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHNH<sub>2</sub> (0.7 g.) in 7 ml. EtOH treated with 1 g. o-, m-, or p-IC<sub>6</sub>H<sub>4</sub>CHO followed by 0.8 ml. AcOH yielded the corresponding hydrazones, o-iodo derivative (IVa), m. 244°, m-iodo derivative (IVb), m. 212°, p-iodo derivative (IVc), m. 201°. Powdered

Ia (1.52 g.) in 8 ml. H<sub>2</sub>O and 3.2 ml. HCl diazotized at 0-5° with 0.72 g. NaNO<sub>2</sub> in 4 ml. H<sub>2</sub>O, the solution allowed to stand about 5 min., added to 3.66 g. of IVa, IVb, or IVc in 80 ml. tetrahydrofuran, followed immediately by 6.0 g. KOH in 20 ml. H<sub>2</sub>O (the temperature during mixing held at -20 to -25°), the mixture allowed to come to room temperature after 2 hrs., diluted with H<sub>2</sub>O, filtered, the precipitate washed with 200 ml. hot MeOH and

H<sub>2</sub>O,

extracted (Soxhlet) with tetrahydrofuran followed by EtOH, the extract concentrated,

and diluted with petr. ether yielded the monoformazans, p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHN:CR<sub>1</sub>N:NR<sub>2</sub> (R<sub>1</sub>, R<sub>2</sub>, m.p., and % yield given): o-IC<sub>6</sub>H<sub>4</sub>, 2,4-MeO(3-MeOC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>3</sub> (V), 205° (decomposition) (shrinks at 200°), 55; m-IC<sub>6</sub>H<sub>4</sub>, 2,4-MeO(3-MeOC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>3</sub> (VI), 163° after shrinking, 65; p-IC<sub>6</sub>H<sub>4</sub>, 2,4-MeO(3-MeOC<sub>6</sub>H<sub>4</sub>)C<sub>6</sub>H<sub>3</sub> (VII), 127°

(decomposition), 45. For other monoformazans, the corresponding entities were:

Ph, p-MeOC<sub>6</sub>H<sub>4</sub> (VIII), 197° (decomposition) (shrinks at 193°), 56; Ph, p-ClC<sub>6</sub>H<sub>4</sub> (IX), 215° (decomposition), 29; Ph, p-BrC<sub>6</sub>H<sub>4</sub> (X), 204-5° (decomposition), 47; Ph, p-HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub> (XI), 217° (decomposition), 28; Ph, p-NCC<sub>6</sub>H<sub>4</sub> (XII), 219-20° (decomposition), 32; Me, p-IC<sub>6</sub>H<sub>4</sub> (XIII), 216° (decomposition), 44. The insol. residue from VI, VII, and VIII recrystd. from pyridine yielded the corresponding diformazans (R, m.p., and % yield given): o-IC<sub>6</sub>H<sub>4</sub> (XIV), 263° (decomposition), 5; m-IC<sub>6</sub>H<sub>4</sub> (XV), -, 18; p-IC<sub>6</sub>H<sub>4</sub> (XVI), 278-9° (decomposition), 32. XIV, XV, and XVI (0.24 g.) in 15 ml. tetrahydrofuran and 15 ml. dioxane containing 0.5 ml. iso-AmNO<sub>2</sub> saturated with dry HCl at 0°, the mixture stirred 4 hrs. at room temperature, filtered with C, and the filtrate concentrated in vacuo yielded the corresponding ditetrazolium chlorides (R, m.p., and % yield given): o-IC<sub>6</sub>H<sub>4</sub>, 197° (decomposition), 78; m-IC<sub>6</sub>H<sub>4</sub>, 156-8° (decomposition), 75; p-IC<sub>6</sub>H<sub>4</sub>, 210° (decomposition), 56. p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NHN:CHPh (2.4 g.) in 25 ml. pyridine cooled to 0°, 1.18 g. p-NCC<sub>6</sub>H<sub>4</sub>NH<sub>2</sub> in 1 ml. AcOH and 3 ml. H<sub>2</sub>O treated with 3 ml. HCl, the mixture diazotized with 0.58 g. NaNO<sub>2</sub> in H<sub>2</sub>O at 0°, the solution filtered, the filtrate added slowly to the preceding solution, the mixture stirred 4 hrs. at 0°, kept overnight at room temperature, diluted with H<sub>2</sub>O, and the precipitate washed with H<sub>2</sub>O yielded XII. XI was prepared in the same manner. IX, X, and XIII were prepared similarly except that a mixture of dioxane and tetrahydrofuran was used as solvent and 10% NaOH was used as the condensing agent. XII (0.15 g.) in 20 ml. tetrahydrofuran treated with 0.2 ml. iso-AmNO<sub>2</sub>, the solution cooled to 0°, saturated with dry HCl, stirred 2 hrs. at room temperature, the solvent removed (steam bath), the residue dissolved in a few drops of EtOH, and diluted with Et<sub>2</sub>O yielded 67% 2-(p-nitrophenyl)-3-[p-cyanophenyl(R<sub>1</sub>)]-5-phenyl-(R<sub>2</sub>)-monotetrazolium chloride, m. 227° (decomposition). The following compds. were prepared in the same manner (R<sub>1</sub>, R<sub>2</sub>, m.p., and % yield given): Ph, p-OMeC<sub>6</sub>H<sub>4</sub>, 187° (decomposition), 69; Ph, p-ClC<sub>6</sub>H<sub>4</sub>, 253° (decomposition), 87; Ph, p-BrC<sub>6</sub>H<sub>4</sub>, 232-4° (decomposition), 83; Ph, HO<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>, 230° (decomposition), 78; Ph, p-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>, 227-9° (darkened at 215°), 83; Me, p-IC<sub>6</sub>H<sub>4</sub>, 175-85°, 66. The series of 3-Ph substituted 2-(p-nitrophenyl)tetrazolium salts were compared with respect to reduction by the dehydrogenase systems in rat-liver homogenates with succinic, lactic, β-hydroxybutyric, and isocitric dehydrogenases. Phenazine methosulfate was used as the intermediate electron carrier for the transfer of electrons from either the reduced enzyme or coenzyme to the tetrazolium salt. With each enzyme, maximum reduction was obtained with 3-(p-iodophenyl)-2-(p-nitrophenyl)tetrazolium chloride (INT). Low activity was observed with 2-(p-nitrophenyl)-3,5-diphenyltetrazolium chloride as compared with INT. When the 3-(p-phenyl) H atom was replaced by groups with increasing electronegativity, such as CO<sub>2</sub>H, Cl, and Br, progressively greater formazan production was noted with each enzyme. Substitution of cyano and nitro groups, which were more electroneg. than iodo, did not result in greater formazan production than was observed with INT. The possibility of steric influences was considered. Replacement of the 5-Ph with a 5-Me group significantly decreased formazan formation by all 4 dehydrogenase systems. This finding emphasized the importance of the 5-Ph group in facilitating the opening of the tetrazolium ring.

L8 ANSWER 4 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1959:119516 CAPLUS

DOCUMENT NUMBER: 53:119516

ORIGINAL REFERENCE NO.: 53:21314h-i,21315a-b

TITLE: Dehydration of hydrazine hydrate by alkaline earth oxides and entrainment by aromatic hydrocarbons  
Pannetier, Guy; Bultingaire-Laborde, Madeleine  
Fac. sci., Paris  
Buletin de la Societe Chimique de France (1958)  
1397-400

CODEN: BSCFAS; ISSN: 0037-8968

DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB Safety standards are defined by which the organic inhibitors studied (except III) may be mixed with I-hydrate (VIII) in the liquid phase in the presence of a desiccant, e.g., CaO, BaO, concentrated NaOH, or concentrated KOH, in proportions such as to assure total entrainment of I in the gas phase with complete freedom from explosive decomposition. For each inhibitor, the min. concentration in the liquid phase which meets this standard is: II 37.63, IV 36.75, V 34.64, VI, 18.95, and VII 22.72%. Several expts. on the dehydration of VIII with CaO by using II as the inhibitor, gave the following over-all results: exptl. yields were reproducible for any given starting proportion of II, VIII, and CaO; highest yields were obtained where the volume in cc. of II was 10 times, and the weight in g. of CaO was 6 times, the volume in cc. of VIII containing 63% I; the purity of II, e.g. reagent grade or tech., did not affect yield, but the purity of CaO was important and had to be as high as possible; by considering the explosive hazard and need for good phys. contact among the 3 phases in the distillation mixture, the proper order of addition was: CaO, II, VIII, with good mech. mixing for at least 1 hr. prior to distillation; distillation at 78-9° yielded a total pressure of I + II in the apparatus of one atmospheric; a slow rate of distillation was essential to good yields; toward the end of the distillation, the temperature had to be raised to 92-6° in order to recover the last traces of both I and II; yields of approx. 95% of 99%-pure I, and approx. 96% of II were obtained, I and II separating in the distillate, with supernatant II protecting I from the atmospheric

L8 ANSWER 5 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1959:39941 CAPLUS  
DOCUMENT NUMBER: 53:39941  
ORIGINAL REFERENCE NO.: 53:7177i, 7178a-i, 7179a-i, 7180a-i, 7181a  
TITLE: Mechanism of chemical reactions. XVIII. Specific catalytic condensations with dihalides. 1. Conversion of aliphatic-aromatic dichlorides into amines with properties of local anesthetics  
AUTHOR(S): Kindler, Karl; Hansen, Werner; Koebke, Jurgen  
CORPORATE SOURCE: Univ. Hamburg, Germany  
SOURCE: Ann. (1958), 617, 25-54  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable  
OTHER SOURCE(S): CASREACT 53:39941

AB cf. C.A. 52, 9015g. PhCH<sub>2</sub>CH<sub>2</sub>OH (980 g.) at 130° was treated dropwise with 1 kg. SOCl<sub>2</sub>, heated another 1.5 hrs., giving 1.012 kg. PhCH<sub>2</sub>CH<sub>2</sub>Cl, b<sub>10</sub> 78-9°, 5 moles of which with 75 g. paraformaldehyde and 75 g. anhydrous ZnCl<sub>2</sub>, stirred 30 min. at 45°, treated 6.5 hrs. with a rapid stream of HCl (keeping a slight pressure for 30 min.), washed repeatedly with saturated aqueous NaCl, and then with aqueous NaCl and NaHCO<sub>3</sub> to insure complete removal of HCl and ZnCl<sub>2</sub>, and fractionated gave 337 g. 4-ClCH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl (I), b<sub>9</sub> 137-8°, m. 34-5° (MeOH-PrOH with traces H<sub>2</sub>O). Similarly, 8.4 moles SOCl<sub>2</sub> and 8 moles Ph(CH<sub>2</sub>)<sub>3</sub>OH gave 1.1 kg. Ph(CH<sub>2</sub>)<sub>3</sub>Cl, b<sub>8</sub> 86-88°, 775 g. of which with 75 g. paraformaldehyde gave 345 g. 4-Cl(CH<sub>2</sub>)<sub>3</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl (II), b<sub>9</sub> 150-2°, n<sub>D</sub><sup>20</sup> 1.5468. I with excess morpholine (III) in xylene, heated 14 hrs. at 125° gave p-(RCH<sub>2</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>R (R = morpholino), b<sub>0.8</sub> 184-6°; dipicrate, m. 190-2°. Similarly III and II gave p-RCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>R, b<sub>0.9</sub> 196-8°; dipicrate, m. 202-3°. These derivs. of III were used in judging the purity of various preps. of I and II. Mesitylene (IV) and other aromatic hydrocarbons and ethers (in excess) were condensed with I or II by using min. amts. of FeCl<sub>2</sub> as catalyst, and passing CO<sub>2</sub>-free air through the mixture to remove HCl. Excessive amts. of FeCl<sub>3</sub> gave greatly decreased yields. The

following monochlorides, p-R'CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>Cl (V) were obtained from 0.1 mole I (other reactant, mg. FeCl<sub>3</sub>, temperature, reaction time in min. R', b.p./mm., and % yield of V given): C<sub>6</sub>H<sub>6</sub>, 90, 90°, 80, Ph, 185-7°/8, 59; IV, 0.06, 115°, 45, 2,4,6-MeC<sub>6</sub>H<sub>2</sub>, 207-9°/8, 78; MeOPh, 60, 110°, 60, 4-(MeO)C<sub>6</sub>H<sub>4</sub>, 212-15°/10, 84; 2-MeC<sub>6</sub>H<sub>4</sub>OMe, 15, 115°, 15, 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>, 229-33°/16, 76; 2-ClC<sub>6</sub>H<sub>4</sub>OMe, 30, 115°, 35, 3,4-Cl(MeO)C<sub>6</sub>H<sub>3</sub>, 215-17°/0.7, 58. Similarly formed from II were the following p-R'CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>Cl (VI) (with similar data given): naphthalene, 67, 100°, 25, α-C<sub>10</sub>H<sub>7</sub>, 221-23°/1.5, 59; 1-methylnaphthalene, 81, 100°, 20, 4-MeC<sub>10</sub>H<sub>6</sub>, 232-4°/1.1, 68; Tetralin, 50, 120°, 100, 5,6,7,8-tetrahydro-β-naphthyl, 214-16°/1.4, 58; IV, 0.17, 115°, 25, 2,4,6-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, 196-99°/1.3, 79; PhMe, 30, 100°, 30, 4-MeC<sub>6</sub>H<sub>4</sub>, 205-8°/10, 60; PhEt, 30, 110°, 40, 4-EtC<sub>6</sub>H<sub>4</sub>, 214-17°/10, 73; PhPr, 15, 115°, 60, 4-PrC<sub>6</sub>H<sub>4</sub>, 180-1°/0.8, 66; 1,4-Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, 30, 110°, 20, 2,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 214-18°/10, 70; 1,2-Me<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, 90, 110°, 90, 3,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 220-23°/10, 73; 4-iso-PrC<sub>6</sub>H<sub>4</sub>, 45, 155-60°, 70, 2,5-Me(iso-Pr)C<sub>6</sub>H<sub>3</sub>, 186-90°/0.8, 70; PhCl, 15, 115°, 35, 4-ClC<sub>6</sub>H<sub>4</sub>, 174-77°/0.8, 22; p-ClC<sub>6</sub>H<sub>4</sub>OH, 4, 100°, 85, 5,2-Cl(HO)C<sub>6</sub>H<sub>3</sub>, 226-30°/1.2, 57; o-ClC<sub>6</sub>H<sub>4</sub>OH, 5, 100°, 3,4-Cl(HO)C<sub>6</sub>H<sub>3</sub>, 90, 232-36°/1.1, 63; MeOPh, 11, 100° 35, 4MeOC<sub>6</sub>H<sub>4</sub>, 185°/1.5, 68; EtOPh, 24, 100°, 30, 4-EtOC<sub>6</sub>H<sub>4</sub>, 208-10°/2.4, 65; PropH, 25, 100°, 65, 4-PrOC<sub>6</sub>H<sub>4</sub>, 214-18°/2.2, 69; BuOPh, 10, 100°, 25, 4-BuOC<sub>6</sub>H<sub>4</sub>, 207-10°/1.2, 59; 2-ClC<sub>6</sub>H<sub>4</sub>OMe, 6, 100°, 20, 3,4-Cl-(MeO)C<sub>6</sub>H<sub>3</sub>, 213°/1.3, 73; 2-ClC<sub>6</sub>H<sub>4</sub>OEt, 7, 100°, 40, 3,4-Cl(EtO)C<sub>6</sub>H<sub>3</sub>, 225-27°/2, 66; 2-ClC<sub>6</sub>H<sub>4</sub>OPr, 18, 130°, 25, 3,4-Cl(PrO)C<sub>6</sub>H<sub>3</sub>, 219-21°/0.8, 66; 2-iso-PrC<sub>6</sub>H<sub>4</sub>Cl, 17, 130°, 30, 3,4-Cl(iso-Pr)C<sub>6</sub>H<sub>3</sub>, 204-6°/0.9, 52; 2-ClC<sub>6</sub>H<sub>4</sub>Bu, 18, 125°, 15, 3,4-Cl(Bu)C<sub>6</sub>H<sub>3</sub>, 238-40°/1.3, 64; 2-Me-C<sub>6</sub>H<sub>4</sub>OMe, 6, 100°, 120, 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>, 200-3°/1.5, 72; 4-MeC<sub>6</sub>H<sub>4</sub>OMe (VII), 23, 110°, 130, 5,2-Me(MeO)C<sub>6</sub>H<sub>3</sub> (VIIa), 204-6°/2.6, 62; 2-MeC<sub>6</sub>H<sub>4</sub>OEt, 15, 100°, 30, 3,4-Me(EtO)C<sub>6</sub>H<sub>3</sub>, 201-3°/0.9, 75; 2-MeC<sub>6</sub>H<sub>4</sub>OPr, 16, 125°, 30, 3,4-Me(PrO)C<sub>6</sub>H<sub>3</sub>, 207-9°/1.3, 66; 2-MeC<sub>6</sub>H<sub>4</sub>OBu, 28, 145°, 65, 3,4-Me(BuO)C<sub>6</sub>H<sub>3</sub>, 217-19°/1.6, 63; 2-MeC<sub>6</sub>H<sub>4</sub>OCH<sub>2</sub>CH<sub>2</sub>OEt, 66, 120°, 170, 3,4-Me(EtOCH<sub>2</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>, 225-32°/1.5, 78; Ph<sub>2</sub>O, 12, 105°, 140, 4-PhOC<sub>6</sub>H<sub>4</sub>, 225-7°/0.7, 65; 1,2(MeO)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, 25, 120°, 25, 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 212-14°/1, 62; 1,3-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>, 11, 110°, 215, 2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 235-7°/4, 74; 1,2,3(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, 18, 130°, 15, 2,3,4-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, 229-31°/1.5, 69; 1-MeO-C<sub>10</sub>H<sub>7</sub>, 9, 100°, 20, 4-MeOC<sub>10</sub>H<sub>6</sub>, 243-6°/1.3, 68; dihydrosafrole 30, 100°, 50, 3,4-(CH<sub>2</sub>O<sub>2</sub>)-6-PrC<sub>6</sub>H<sub>2</sub>, 214-19°/0.8, 62; dihydroeugenol Me ether, 30, 110°, 65, 3,4,6(MeO)<sub>2</sub>PrC<sub>6</sub>H<sub>2</sub>, 205-8°/0.4, 69; dihydroanethole, 30, 110°, 35, 2,5-(MeO)PrC<sub>6</sub>H<sub>3</sub>, 191-4°/0.6, 61. In forming the following VI, from 0.15-0.2 mole II, anhydrous AlCl<sub>3</sub> or ZnCl<sub>2</sub> was used (reactant, mg. of catalyst, temperature and reaction period in min, R', b.p./mm. and yield given): C<sub>6</sub>H<sub>6</sub>, 105 AlCl<sub>3</sub>, 90°, 120, Ph, 191-2°/6, 42; PhF, 193 AlCl<sub>3</sub>, 90°, 200, 4-FC<sub>6</sub>H<sub>4</sub>, 180-3°/3, 25; PhCl, 147 AlCl<sub>3</sub>, 105°, 60, 4-ClC<sub>6</sub>H<sub>4</sub>, 203-4°/2.7, 20; PhBr, 118 AlCl<sub>3</sub>, 110°, 60, 4-BrC<sub>6</sub>H<sub>4</sub>, 218-20°/3.5, 17; VII, 42 AlCl<sub>3</sub>, 110°, 90, VIIa, 198-200°/2.6, 83; Ph<sub>2</sub>O 9 AlCl<sub>3</sub>, 105°, 80, 4-PhOC<sub>6</sub>H<sub>4</sub>, 247-9°/2, 63; MeOPh, 12 ZnCl<sub>2</sub>, 100°, 70, 4-MeOC<sub>6</sub>H<sub>4</sub>, 209-11°/2.6, 70; VII, 32 ZnCl<sub>2</sub>, 110°, 35, VIIa, 198-200°/2.6, 74. V and VI were purified by repeated distns.; no analyses are given. V and VI were condensed with primary or secondary amines in Et<sub>3</sub>N or xylene, usually by heating 16-20 hrs. at 125°, using sealed tubes when required. After removal of the solvent and excess amine, the products were frequently extracted with Et<sub>2</sub>O converted into the HCl salts, which after washing with Et<sub>2</sub>O were reconverted into the free bases and extracted with Et<sub>2</sub>O. The exts. were washed with H<sub>2</sub>O and recoveries were obtained both from the Et<sub>2</sub>O exts. and

H<sub>2</sub>O-washings. From V the products were p-RCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>R' (VIII) (R' = substituted amino group). V(R = Ph) with MeNH<sub>2</sub> in Et<sub>3</sub>N gave 79% VIII (R = Ph, R' = NHMe), b<sub>1</sub> 149-51; HCl salt, m. 191-2°: picrate, m. 95-6°. Similarly formed from the appropriate V and III in xylene were 92% VIII (R = 4-MeOC<sub>6</sub>H<sub>4</sub>, R' = morpholino), b<sub>1</sub> 218-20°, and 89% VIII (R = 2,4,6-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>; R' = morpholino), b<sub>1</sub> 220-2°, n<sub>20</sub>D 1.5634. Similarly from the appropriate V and amine were formed 63% VIII (R = 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>, R' = Et<sub>2</sub>N), b<sub>0.3</sub> 175-8°, n<sub>20</sub>D 1.5458, and 76% VIII (R = 3,4-Cl(MeO)C<sub>6</sub>H<sub>3</sub>, R' = morpholino), b<sub>2</sub> 242-6°. Analogous condensation, usually under similar conditions, were effected with the appropriate VI and various amines giving the following compds. p-R'CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>CH<sub>2</sub>R' (IX) (R', R'', b.p./mm. % yield and n<sub>20</sub>D given): α-Cl<sub>10</sub>H<sub>7</sub>, morpholino, 252-4°/1.8, 89, -; 4-MeC<sub>10</sub>H<sub>6</sub>, morpholino, 260-2°/0.8, 92, -; 5,6,7,8-tetrahydro-β-naphthyl, morpholino, 243-6°/1.6, 94, -; tetrahydro-β-naphthyl, Et<sub>2</sub>N, 208-11°/0.6, 77, 1.5569; 2,4,6-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, morpholino, 233-5°/1.7, 92, -; 4-MeC<sub>6</sub>H<sub>4</sub>, morpholino, 193-5°/0.4, 82, 1.5582; 4-MeC<sub>6</sub>H<sub>4</sub>, Et<sub>2</sub>N, 173-5°/0.6, 73, 1.5401; 4-EtC<sub>6</sub>H<sub>4</sub>, morpholino, 203-6°/0.5, 83, 1.5548; 4-EtC<sub>6</sub>H<sub>4</sub>, Et<sub>2</sub>N, 175-8°/0.5, 72, 1.5381; 4-PrC<sub>6</sub>H<sub>4</sub>, Et<sub>2</sub>N, 194-7°/0.9, 65, 1.5338; 4-PrC<sub>6</sub>H<sub>4</sub>, morpholino, 216-19°/0.9, 72, 1.5504; 2,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, morpholino, 208-11°/0.5, 88, 1.5581; 2,5-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, Et<sub>2</sub>N, 174-77°/0.5, 71, 1.5407; 3,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, morpholino, 204-7°/0.4, 84, 1.5589; 3,4-Me<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, Et<sub>2</sub>N, 181-4°/0.6, 67, 1.5417; 2,5-Me(iso-Pr)C<sub>6</sub>H<sub>3</sub>, morpholino, 243-5°/1.7, 83, -; 4-ClC<sub>6</sub>H<sub>4</sub>, Et<sub>2</sub>N, 190-2°/0.6, 1.5493; 5,2-Cl(HO)C<sub>6</sub>H<sub>3</sub>, morpholino, 270-1°/2.5 (with subsequent crystallization), 85, -; 3,4-Cl(HO)C<sub>6</sub>H<sub>3</sub>, morpholino, 260-4°/0.7 (with crystallization), 81, -; 4-MeOC<sub>6</sub>H<sub>4</sub>, morpholino, 239-40°/3, 92, -; 4-MeOC<sub>6</sub>H<sub>4</sub>, N-cyclohexyl methylamino, 242-4°/2, 78, -; 4-MeOC<sub>6</sub>H<sub>4</sub>, (CH<sub>2</sub>:CHCH<sub>2</sub>)<sub>2</sub>N, 232°/0.5, 78, -; 4-MeOC<sub>6</sub>H<sub>4</sub>, Pr<sub>2</sub>N, 213-15°/1.3, 60, -; 4-MeOC<sub>6</sub>H<sub>4</sub>, pyrrolidino, 206-8°/1, 93°, -; 4-EtOC<sub>6</sub>H<sub>4</sub>, morpholino, 232-4°/1.2, 89, -; 4-PrOC<sub>6</sub>H<sub>4</sub>, morpholino, 235-7°/1, 87, -; 4-BuOC<sub>6</sub>H<sub>4</sub>, morpholino, 254-6°/1.7, 88, -; 3,4-Cl(MeO)C<sub>6</sub>H<sub>3</sub>, morpholino, 242°/1.2, 86, -; 3,4-Cl(MeO)C<sub>6</sub>H<sub>3</sub>, MeNBu, 226-8°/1, 50, -; 3,4-Cl(EtO)C<sub>6</sub>H<sub>3</sub>, morpholino, 3,4-Cl(PrO)C<sub>6</sub>H<sub>3</sub>, morpholino, 250-2°/1.1, 87, -; 3,4-Cl(iso-PrO)C<sub>6</sub>H<sub>3</sub>, morpholino, 249-51°/1.4, 54, -; 3,4-Cl(BuO)C<sub>6</sub>H<sub>3</sub>, morpholino, 246-8°/1, 87, -; 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>, morpholino, 226-8°/1, 89, -; in the following VIII. R = 3,4-Me(MeO)C<sub>6</sub>H<sub>4</sub>, and only R', b.p./mm. and % yields are given: Et<sub>2</sub>N, 208°/0.4, 68, EtBuN, 206-9°/0.5, 76; Bu<sub>2</sub>N, 244-7°/0.7, 57; (CH<sub>2</sub>:CHCH<sub>2</sub>)<sub>2</sub>N, 230-2°/0.6, 80; 2-methyl-1-piperidyl, 229-32°/1, 83; 2,5-dimethyl-1-piperazinyl, 239-42°/1, 41. In the following VIII, R and R' b.p./mm. and yields are given: 5,2-Me(MeO)C<sub>6</sub>H<sub>3</sub>, morpholino, 234-7°/1.1, 85; 3,4-Me(EtO)C<sub>6</sub>H<sub>3</sub>, morpholino, 221-23°/0.7, 91; 3,4-Me(EtO)C<sub>6</sub>H<sub>3</sub>, Et<sub>2</sub>N, 193-7°/0.6, 84, n<sub>20</sub>D 1.5362; 3,4-Me(PrO)C<sub>6</sub>H<sub>3</sub>, morpholino, 247-9°/1.3, 86; 3,4-Me(PrO)C<sub>6</sub>H<sub>3</sub> Et<sub>2</sub>N, 195-8°, 67, n<sub>20</sub>D 1.5323; 3,4-Me(BuO)C<sub>6</sub>H<sub>3</sub>, morpholino, 244-6°/1.5, 93; 3,4-Me(EtOCH<sub>2</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>, morpholino, 253-7°/1.2, 75; 4-PhOC<sub>6</sub>H<sub>4</sub>, morpholino, 263-5°/1.1, 87; 3,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, morpholino, 232-4°/0.6, 88; 2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>, morpholino, 232-3°/0.8, 89; 2,3,4-(MeO)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>, morpholino, 246-7°/1.5, 87; 4-MeOC<sub>10</sub>H<sub>6</sub>, morpholino, 268-70°/0.8, 85; 3,4-(CH<sub>2</sub>O)<sub>2</sub>6-PrC<sub>6</sub>H<sub>2</sub>, morpholino, 242-4°/0.6, 61, n<sub>20</sub>D 1.5601; 3,4-(CH<sub>2</sub>O)<sub>2</sub>6-PrC<sub>6</sub>H<sub>2</sub>, Et<sub>2</sub>N, 206-9°/0.5, 62, n<sub>20</sub>D 1.5451; 3,4,6-(MeO)<sub>2</sub>PrC<sub>6</sub>H<sub>2</sub>, morpholino, 245-7°/0.7, 86, n<sub>20</sub>D 1.5538; 3,4,6-(MeO)<sub>2</sub>PrC<sub>6</sub>H<sub>2</sub>, Et<sub>2</sub>N, 209-12°/0.6, 78, n<sub>20</sub>D 1.5382; 2,5-(MeO)PrC<sub>6</sub>H<sub>3</sub>, morpholino, 230-2°/0.6, 87, n<sub>20</sub>D 1.5510; 2,5-(MeO)PrC<sub>6</sub>H<sub>3</sub>, Et<sub>2</sub>N, 190-2°/0.4, 71, n<sub>20</sub>D 1.5357; 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>, MeOCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NH, 219-22°/0.7, 84, n<sub>20</sub>D 1.5450. Na (0.1 g. atom) powdered by heating in 40 cc. boiling xylene was treated with a suitable amino alc. (usually β-morpholinoethanol (IX) in 10 cc. xylene, and heated 15 hrs. at 150° with the appropriate VI. The mixture was treated with Et<sub>2</sub>O and the organic phase washed with H<sub>2</sub>O, shaken with 6% HCl,

and the resulting salt reconverted into the free ether with KOH, extracted with Et<sub>2</sub>O, washed and distilled to give the following p-derivs. of γ-phenylpropyl β-morpholinoethyl ether (p-substituent, b.p./mm., and % yield given): α-naphthylmethyl, 272°/1, 72;

4-FC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, 236-9°/1.8, 81; 4-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, 242-4°/0.8, 82;

3,4-Cl(PrO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 284-6°/2, 79; 3,4-Cl(BuO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 276-82°/1.0-1.3, 77; 3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 258-60°/1.5, 85;

3,4-Me(EtO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 253-6°/0.9, 70; 3,4-Me(PrO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 262-4°/1.5, 67; 3,4-Me(EtOCH<sub>2</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 274-7°/1.5, 61;

PhOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>, 297-99°/0.7, 72; 2,4-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>, 262-4°/1.0, 60. Formed analogously were the following [p-(4-methoxybenzyl)-γ-

phenylpropyl]-β-piperidinoethyl ether, 227-9°/0.7, 75;

[p-(4-methoxy-3-methylbenzyl)-γ-phenylpropyl]-β-dibutylaminoethyl ether, 279-82°/1.1, 53; and [p-(2-methoxy-5-

methylbenzyl)-γ-phenylpropyl]-2-morpholinocyclohexyl ether, 276-9°/1.3, 40. 2-O<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>OMe (X) (0.6 mole) heated 90 min. at 115° with 0.2 mole 4-[Cl(CH<sub>2</sub>)<sub>3</sub>]C<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl and 90 mg. FeCl<sub>3</sub> gave

p-(4-methoxy-3-nitrobenzyl)-γ-phenylpropyl chloride (XI) which could not be distd; after removing excess X, 67 g. crude XI was treated with

52.3 g. III to give 49 g. crude, undistillable N-[(p-4-methoxy-3-nitrobenzyl)-γ-phenyl] morpholine, (XII), 18.5 g. of which, in 150

cc. MeOH was hydrogenated 8 hrs. with 10 g. moist Raney Ni at 2.5 atmospheric, filtered, treated with 6% HCl, and processed as above, to yield 53% 3-NH<sub>2</sub>

analog of XII, C<sub>21</sub>H<sub>28</sub>N<sub>2</sub>O<sub>2</sub>, b<sub>0.7</sub> 250-3°/1, m. 67-8°.

p-[5,2-Me(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>]C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>3</sub>Cl (XIII) (0.2 mole), heated and stirred 22 hrs. with 0.24 mole NaI in 200 cc. PrOH, evaporated, treated with H<sub>2</sub>O and

the resulting crude iodide analog (XIIIa) of XIII was filtered; the aqueous solution was extracted repeatedly with Et<sub>2</sub>O, and these evaporated exts. were

combined

with XIIIa, b<sub>12</sub> 212-28° (total yield 78%). XIIIa (53.8 g.) with 18.2 g. KCN in 250 cc. 80% PrOH, stirred and refluxed 20 hrs., evaporated,

dissolved in H<sub>2</sub>O and extracted with Et<sub>2</sub>O gave 33.2 g. crude p-(2-methoxy-5-methylbenzyl)-γ-phenylbutyrylonitrile (XIV), freed

from residual XIIIa by heating with III in xylene and extracting with 6% HCl. Purified XIV, b<sub>1.5</sub> 202-9°. XIV (9.3 g.) hydrogenated and

shaken with 20 g. moist Raney Ni in MeOH saturated with NH<sub>3</sub>, filtered, evaporated

dissolved in dilute HCl, washed with Et<sub>2</sub>O, and made alkaline with KOH gave 5 g. p-(5,2-Me(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>4</sub>NH<sub>2</sub>, (XV), b<sub>0.8</sub> 194-7°. Formed

analogously to XV with only slight modifications from the appropriate nitrile was p-(3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>4</sub>NH<sub>2</sub>, very hygroscopic, b<sub>1</sub> 208-11°. A mixture of 14 g. p-(3,4-Me(MeO)C<sub>6</sub>H<sub>3</sub>CH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>3</sub>CN, 13

g. III and 50 cc. MeOH was hydrogenated at 20° with Raney Ni as above to give 6.9 g. crude N-[p-(3-methyl-4-methoxybenzyl)-δ-

phenylbutyl]morpholine (XVI), purified by treating in Et<sub>2</sub>O with BzCl and aqueous NaOH, separating the Et<sub>2</sub>O phase, treating with dilute HCl, and freeing

the

base with KOH to give 5.0 g. XVI, b<sub>1.5</sub> 242-5°; picrolonate, m. 210°. PhCH<sub>2</sub>Cl (0.1 mole) and 0.6 mole MeOPh (XVII) or 2-MeC<sub>6</sub>H<sub>4</sub>OMe

and small amts. of FeCl<sub>3</sub> reacted readily at 100° giving the usual type of condensation products (e.g. PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>Me) in yields of 80-87%; with

large amts. of FeCl<sub>3</sub> yields decreased, and in the absence of catalyst no condensations occurred. Mesitylene reacted readily with 0.1 mole

p-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl and 0.012 mg. FeCl<sub>3</sub>. Ph(CH<sub>2</sub>)<sub>2</sub>Cl or Ph(CH<sub>2</sub>)<sub>3</sub>Cl, even in the presence of small amts. of FeCl<sub>3</sub> failed to react with XVII. With large

amts. of FeCl<sub>3</sub>, at 150°, HCl was liberated, but no identifiable products were isolated. In the presence of 0.0005 mole of certain

inhibitors (notably substituted thiobenzamides and (Et<sub>2</sub>N)<sub>2</sub>CO), the yields of PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OMe were decreased greatly, unless much larger amts. of FeCl<sub>3</sub>

were used. E.g. in the presence of 0.0005 mole N-thiobenzoylpiperidine (XVIII), the amount of FeCl<sub>3</sub> had to be increased from the usual 1 mg. to 81

mg. to give a satisfactory yield of PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OMe. Whereas 0.3 mole XVII, 0.1 mole PhCH<sub>2</sub>Cl, and 1 mg. FeCl<sub>3</sub> at 115° gave 70% PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OMe in

30 min., the introduction initially of 0.1 g. XVIII gave rise to neither HCl nor PhCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>OMe. Neither diethyl- nor phenylethylbarbituric acid

served as inhibitors. PhCH<sub>2</sub>Cl (25.4 g.) and 60 mg. FeCl<sub>3</sub> in PhNO<sub>2</sub> reacted violently giving HCl and a resin, however, when 0.5 g. Me(CH<sub>2</sub>)<sub>4</sub>CONH<sub>2</sub> or Me(CH<sub>2</sub>)<sub>16</sub>CONH<sub>2</sub> was added to the original mixture no HCl was formed, even on heating several hrs., and PhCH<sub>2</sub>Cl was recovered almost quantitatively. Similar results were obtained when these amides were added to other arylmethyl chlorides, I, or II, even in the presence of such resin-forming catalysts as ZnCl<sub>2</sub> or AlCl<sub>3</sub>. The statement is made that the above condensations (giving amines) can yield local anesthetics; no pharmacol. data are given.

L8 ANSWER 6 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1951:57821 CAPLUS  
DOCUMENT NUMBER: 45:57821  
ORIGINAL REFERENCE NO.: 45:9842d-f  
TITLE: New Arosorb process  
AUTHOR(S): Weber, George  
CORPORATE SOURCE: Petroleum Publishing Co., New York, NY  
SOURCE: Oil & Gas Journal (1951), 50(No. 11), 58-9  
CODEN: OIGJAV; ISSN: 0030-1388  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB Sun Oil Co.'s new Arosorb process consists in separating aromatic hydrocarbons produced in catalytic reforming processes by means of selective adsorption of the aromatic hydrocarbons on silica gel. Nonaromatic hydrocarbons pass through the bed and residual nonaromatic hydrocarbons are displaced from the column with butane before the aromatic hydrocarbons are desorbed, thus completing the cycle. The first com. unit will process reformat produced in a 10,000 barrel per day Houdriforming unit now under construction to concentrate and purify annually 13,000,000 gal. of benzene, 30,000,000 gal. of toluene, and 15,000,000 gal. of mixed xylenes. The desorbent in this case will be mixed xylenes. The com. unit will consist of six gel cases containing a total of 500,000 lb. of gel. Gel life will be about 1 yr. based on pilot-plant studies. Each cycle will require about 100 min. Purity of the recovered aromatic hydrocarbons is related to yield as follows: 98% purity = 90% yield, 90% purity = 98% yield. A light H<sub>2</sub>SO<sub>4</sub> treat is required to remove traces of olefins from the benzene and toluene

L8 ANSWER 7 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1940:43218 CAPLUS  
DOCUMENT NUMBER: 34:43218  
ORIGINAL REFERENCE NO.: 34:6594c-i, 6595a-i, 6596a  
TITLE: Characterization of carboxylic acids as ureides with the aid of carbodiimides. IX. Preparation of carbodiimides from thioureas  
AUTHOR(S): Zetzsche, Fritz; Nerger, Wilhelm  
SOURCE: Berichte der Deutschen Chemischen Gesellschaft [Abteilung] B: Abhandlungen (1940), 73B, 467-77  
CODEN: BDCBAD; ISSN: 0365-9488  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB The method of Schmidt and coworkers (C. A. 34, 3687.3) of preparing carbodiimides from thioureas by desulfurization at room temperature and in ether

and CS<sub>2</sub> as well as in benzene, although it is a material improvement over the original Weith method (Ber. 7, 10(1874)) with yellow HgO at elevated temps. (boiling benzene, toluene, xylene), is limited in its applicability by the solubility of the thiourea in the solvent at room temperature. A modification of the Weith method applicable to difficultly soluble

aromatic thioureas was therefore sought. In the system thiourea-carbodiimide-urea-water-metal oxide-metal sulfide, 5 reactions are possible: (1) desulfurization of the thiourea to carbodiimide, metal sulfide and water (primary reaction); (2) addition of water to the carbodiimide to form urea;

(3) reaction of the carbodiimide with the thiourea to form isothiocyanate and guanidine; (4) reaction of the carbodiimide with urea to form isocyanate and guanidine; (5) conversion of the carbodiimide into other (polymerization) products. Hitherto, reaction (2) is the one which has given most trouble. Z. and N. find, however, that the crude aromatic carbodiimides prepared by the Weith method have a more or less pronounced anise-like odor, due to the presence of the corresponding isothiocyanates, which can easily be isolated in 0.1-2% yield by steam distillation. As regards reaction (5) it had previously been found that, under otherwise identical conditions, the yield of (p-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>N:)<sub>2</sub>C decreases with increasing temperature and that of resinous product increases. If these side reactions depend only on time and temperature it would seem that they might be avoided by increasing the desulfurization velocity by increasing the reactive surface of the metal oxide and by the use of a catalyst. It had already been found that HgO can be replaced by PbO. Z. and N. now find that the use of highly dispersed "Tegoglatte" with a particle size of 1 μ instead of ordinary PbO results in the expected increase in reaction velocity and that the addition of S further increases it, so that desulfurization in relatively low-boiling solvents (acetone, MeOH, EtOH, MeCOEt) requires only a few min. instead of several hrs. To avoid greatly varying yields it is necessary, however, to employ the purest possible thioureas. In reaction (1) the carbodiimide and water are formed at the surface of the metal oxide, a part of which is converted into the sulfide. Of the products, only the carbodiimide is soluble in benzene hydrocarbons, CS<sub>2</sub>, CHCl<sub>3</sub>, CCl<sub>4</sub> and ether, while the water adheres to the metal oxide and sulfide particles. Newly formed mols. of carbodiimide therefore encounter a progressively increasing excess of water and hence the possibility of their undergoing reaction (2) increases. The use of alcs. or ketones as solvent should prevent this harmful accumulation of water on the oxide-sulfide surfaces. Comparative expts. with 10 g. (p-MeC<sub>6</sub>H<sub>4</sub>NH)<sub>2</sub>CS, 15 g. "Tegoglatte" and varying amts. of S allowed to react 30 min. in 400 cc. acetone gave the following results: the 3 values given are, resp., the amount of S added (mg.), the yield of crude carbodiimide (g.), and the residue of thiourea or urea (g.): 0, 1.9, 7.55 thiourea; 20, 7.9, 0.95 thiourea; 22, 8.05, 0.45 urea; 25, 7.6, 1.05 urea; 30, 7.8, 1.1 urea; 40, 7.85, 1.2 urea; 70, 8.05, 0.4 urea; 100, 8.1, 0.6 urea; 200, 8.1, 0.45 urea. Similar results were obtained with (p-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH)<sub>2</sub>CS. In both series the yield of carbodiimide was increased by small amts. of S, and as the amount of S was increased the yield of carbodiimide increased to a maximum, then decreased somewhat and finally rose continuously until it approached 100%. Hence the S both catalyzes the primary reaction (1) and inhibits at least one (urea formation) of the secondary reactions. It might be expected that with small amts. of S (1-20 mg.) some urea would be found along with the carbodiimide and unchanged thiourea. That this is not the case indicates that in the absence of large amts. of S its inhibitory action on urea formation is taken over by the thiourea in the early stages of the reaction. With moderate amts. of S (20-50 mg.), after all the thiourea has reacted there is not enough S present to inhibit urea formation and hence if the reaction is not stopped at the proper time all of the carbodiimide may easily be converted into urea. PbO and especially the highly dispersed Tegoglatte, as also, but to a lower degree, PbS favor urea formation in the presence of small amts. of added water, but S hinders this reaction also. For practical reasons an excess of PbO has to be used but it should be limited to about 50% more than the theor. required amount. Acetone proved to be the only satisfactory solvent. Se seems to have the same effect as S. "Tegomennige" gives results similar to those obtained with "Tegoglatte" but the reaction is somewhat more sluggish as the PbO<sub>2</sub> present in the former does not react. Water favors the desulfurization but it also favors the urea formation. PbS and ordinary PbO produce no or very little resinification whereas "Tegoglatte" gives a greatly resinified product, but S hinders this resinification also. By utilizing these observations it is possible to obtain at least 90% yields of carbodiimides in a high state of purity (95%) as determined by the oxalic acid method. The method is wholly inapplicable, however, to thioureas which contain aliphatic or hydrocyclic substituents



(phenylallylthiourea is an exception). Difficultly soluble thioureas, like (p-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>NH)<sub>2</sub>CS, m. 184-6°, are purified by shaking twice for 24 h. with a large volume of pure acetone, more-soluble thioureas by repeated crystallization from absolute alc. A typical carbodiimide synthesis by this method is the following: Synthesis of carbodi(p-tolylimide) on a small scale. In a 1-l. flask provided with a stirrer, condenser and wide (2 cm.) side tube, 10 g. of the thiourea, 200 mg. powdered S and 400 cc. acetone are boiled 20 min., then, irres. of whether or not the thiourea is completely dissolved, 15 g. "Tegoglatte" is added through the side tube; after 15 min., the mixture is filtered, the filtrate is concentrated, treated with 50 cc. petr. ether, allowed to stand 0.5 h. in ice for complete precipitation of the urea or thiourea, filtered, evaporated in a round-bottomed flask and freed from the last traces of solvent by evacuating and shaking, whereupon the carbodiimide suddenly crystallizes. After standing overnight in an open flask it is recrystd. from petr. ether; yield, over 90%. Synthesis on a larger scale. To avoid the necessity of using an unduly large volume of acetone, a smaller proportion of S is used and the desulfurization is stopped at the proper stage (determined by preliminary expts. with small amts.). The thiourea (40 g.) and 0.4 g. S in 400 cc. acetone are brought to a boil, 60 g. "Tegoglatte" is added, the reaction is stopped after 5-8 min., the contents of the flask are at once thoroughly drained and the filtrate is treated as above (yield, 32 g.). (p-Me<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>N)<sub>2</sub>C, m. 88-90°, was similarly prepared Carbodi(4-diethylaminophenylimide) (90% yield), m. 81-2°, is converted by solution in a little dilute HCl and addition to dilute Na<sub>2</sub>CO<sub>3</sub> into the urea, m. 223-4° (corrected). PhNHCSNHC<sub>6</sub>H<sub>4</sub>N:NPh, obtained in 85% yield from p-H<sub>2</sub>NC<sub>6</sub>H<sub>4</sub>N:NPh and PhNCS allowed to stand several weeks in ether, m. 158-9°, resolidifies 160-4° and again m. 168°; 20 g. yields 10.5 g. N-phenyl-N'-phenylazophenylcarbodiimide, light orange-red, m. 60-4°. From the mother liquor is obtained 1.5 g. of the light yellow urea, m. 216°; N-Bz derivative, C<sub>26</sub>H<sub>20</sub>O<sub>2</sub>N<sub>4</sub> (0.65 g. from 600 mg. carbodiimide and 240 mg. BzOH allowed to stand overnight in ether), orange-yellow, m. 117-18°, resolidifies 122° and again m. 156-7°; N-cinnamoyl derivative, orange-yellow, m. 117-18°, partially solidifies 130° and again m. 140°.

L8 ANSWER 8 OF 8 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1907:1680 CAPLUS  
 DOCUMENT NUMBER: 1:1680  
 ORIGINAL REFERENCE NO.: 1:424i,425a-i,426a  
 TITLE: Benzoyl Nitrate, a New Nitrating Agent  
 AUTHOR(S): Francis, Francis  
 CORPORATE SOURCE: University College, Bristol, UK  
 SOURCE: Berichte der Deutschen Chemischen Gesellschaft (1907),  
 39, 3798-3804  
 CODEN: BDCGAS; ISSN: 0365-9496  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable

AB The preparation of benzoyl nitrate, BzONO<sub>3</sub>, from benzoyl chloride and silver nitrate has been previously described. (J. Chemical Society, 89, 1); in order to secure a good yield it is necessary to have the silver nitrate dry, finely divided and to employ 1.2 mols. of it. Benzoyl nitrate is very sensitive to moisture. It explodes with ordinary filter paper, but may be filtered through dry paper. At 0° sp. gr.=1.3, but the purest preparations appear to contain 15-20% of benzoic anhydride. In sealed tubes, at the ordinary temperature, the nitrate gradually solidifies forming benzoic anhydride and, probably, nitric anhydride thus resembling the organic mixed anhydrides. At 100° the change to benzoic anhydride, oxygen and nitrogen dioxide is rapid and at higher temperatures explosive. Only the smallest trace of m-nitrobenzoic acid is formed under the above conditions, but in nitrobenzene solution the change to this substance is fairly quick, 6% in six months. No isomeric acids

could be detected. The change occurs less readily in other indifferent solvents and is very slow in carbon tetrachloride which, therefore, is used in some of the nitration experiments. Other substances of the type of benzoyl nitrate are doubtless formed as intermediate compounds in the course of the action of acid chlorides on metallic nitrates as studied by Lachowicz (Ber., 17, 1281), but their isolation is only possible if the chloride remains liquid at a temperature below that of decomposition of the acyl nitrate. The author has succeeded in preparing metanitrobenzoyl nitrate,  $\text{O}_2\text{NC}_6\text{H}_4\text{COONO}_2$ , and butyryl nitrate,  $\text{MeCH}_2\text{CH}_2\text{COONO}_2$ . The former resembles benzoyl nitrate, m.  $40^\circ\text{--}50^\circ$ . In scaled tubes, or in solution in nitrobenzene it forms m-nitrobenzoic anhydride; no evidence of molecular rearrangement could be detected. Butyryl nitrate, pale yellow liquid, explodes when heated. Benzoyl nitrate readily reacts with thiophene to form nitrothiophene in excellent yield and purity. Ethyl alcohol yields ethyl nitrate. With primary aromatic amines it behaves like benzoyl chloride; secondary aromatic amines yield nitramines whereas the corresponding aliphatic compounds give substituted benzamides. Benzene, toluene, m-xylene and mesitylene form mononitro derivatives the more easily the higher the molecular weight of the hydrocarbon. Brombenzene, benzoyl chloride and benzoyl cyanide react with difficulty, forming small quantities of the p-nitroderivatives. Phenol gives a mixture of o- and p-compounds, anisole and phenetole yield exclusively the ortho derivatives. Veratrole forms the 3-nitro compound. With the naphthols, even in dilute solution, at low temperatures, much decomposition occurs but 2,4-dinitro- $\alpha$ -naphthol and 1,6-dinitro- $\beta$ -naphthol were isolated in small quantity. The corresponding ethers, on the other hand, react smoothly giving 4-nitro- $\alpha$ -naphthol ethyl ether, together with traces of the 1,2-compound, and 1-nitro- $\beta$ -naphthol ethyl and methyl ethers, respectively. Benzaldehyde does not interact with benzoyl nitrate at low temperatures, at higher ones it is slightly oxidized. Anisaldehyde is quickly converted into the acid, a small quantity of a nitro compound being also formed. Salicylic aldehyde is chiefly oxidized, but in presence of carbon tetrachloride 3-nitrosalicylic aldehyde is produced to a limited extent. Orthomethoxy-benzaldehyde is also oxidized. Mandelic nitrile appears to yield the compound,  $\text{PhC}(\text{NO}_2)(\text{OH})\text{CN}$  m.  $77^\circ\text{--}78^\circ$ . It is decomposed by water or alkalis into benzoic acid, nitrous acid and hydrogen cyanide. Vanillin yields 3-nitrovanillin, quantitatively. Piperonal is chiefly oxidized, but a little of the 6-nitro derivative is formed.  $\beta$ -Naphthol aldehyde yields, quantitatively, a dinitro compound, m.  $194^\circ\text{--}195^\circ$  and cumarin gives the 5-nitro derivative. The oxidizing power of benzoyl nitrate has not, as yet, been very extensively investigated. Thiophenol is converted quantitatively into diphenyl disulphide and hydrazobenzene yields azobenzene. Pictet and Genequand's diacetyl-o-nitric acid,  $(\text{AcO})_2\text{N}(\text{OH})_2$ , (Ber., 35, 2526) gives mononitro derivatives of  $\alpha$ - and  $\beta$ -naphthol, paranitraniline from acetanilide and oxidizes aniline, whereas benzoyl nitrate gives o-acetnitraniline and benzanilide, respectively. These differences are ascribed to the fact that benzoyl nitrate is the only substance known which admits of nitration with complete exclusion of moisture. (Cf. following abstract).

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(FILE 'HOME' ENTERED AT 14:16:10 ON 11 MAR 2007)

FILE 'CAPLUS' ENTERED AT 14:26:12 ON 11 MAR 2007

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L1      377 S XYLENE AND TOLUENE AND IMPURITY
L2      0 S L1 AND TRACES OF XYLEN?
L3      0 S L1 AND %
L4      0 S L1 AND COMMERCIAL TOLUENE
L5      5 S L1 AND TECHNICAL
L6      0 S L1 AND TRACES OF XYLENE
L7      12 S TRACES OF XYLENE

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L8 8 S TOLUENE AND PURITY AND TRACES AND XYLEN?

=> S L1 NOT L5

L9 372 L1 NOT L5

=> S L9 NOT L7

L10 372 L9 NOT L7

=> S L10 NOT L8

L11 372 L10 NOT L8

=> S L11 AND XYLENES

7572 XYLENES

L12 30 L11 AND XYLENES

=> D 1-30 IBIB ABS

L12 ANSWER 1 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:623486 CAPLUS

DOCUMENT NUMBER: 143:378730

TITLE: Analysis of benzene, toluene and  
xylenes by capillary gas chromatography

AUTHOR(S): Qin, Jin-ping; Yu, Hong-mei; Tang, Ji-hai

CORPORATE SOURCE: College of Chemistry and Chemical Engineering, Nanjing  
University of Technology, Nanjing, 210009, Peop. Rep.  
China

SOURCE: Nanjing Gongye Daxue Xuebao, Ziran Kexueban (2005),  
27(3), 58-60

CODEN: NGDXAX; ISSN: 1671-7643

PUBLISHER: Nanjing Gongye Daxue Xuebao Bianjibu

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

AB A method was developed for separation and determination of benzene, toluene,  
m-xylene and o-xylene using 0.25 mm + 30 m  
+ 0.5 µm high efficiency quartz capillary column with PEG-20M  
(polyethylene glycol-20000) stationary phase. In the benzene hydrocarbon  
there are some nonarom. hydrocarbon impurities. If polar  
capillary column was preferred, the retention time of aromatic hydrocarbon  
delays greatly and that of nonarom. hydrocarbon shortens. The three  
xylene isomers were separated clearly. This method is obviously  
superior to the GB/T3144-82. The advantage of this method lies in its  
high speed and good separation

L12 ANSWER 2 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:431713 CAPLUS

DOCUMENT NUMBER: 141:111898

TITLE: Thermal Conductivity and Thermal Diffusivity of  
Twenty-Nine Liquids: Alkenes, Cyclic (Alkanes,  
Alkenes, Alkadienes, Aromatics), and Deuterated  
Hydrocarbons

AUTHOR(S): Watanabe, Hideo; Kato, Hideyuki

CORPORATE SOURCE: National Metrology Institute of Japan, AIST, Tsukuba  
Ibaraki, 305-8563, Japan

SOURCE: Journal of Chemical and Engineering Data (2004),  
49(4), 809-825

CODEN: JCEAAX; ISSN: 0021-9568

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Exptl. values of the thermal conductivity  $\lambda$  and thermal diffusivity  $a$  of  
29 pure substances are presented in the temperature range from -15 to +.65  
°C under atmospheric or saturation pressure. The materials measured were as  
follows: alkenes (1-pentene, 1-hexene, 1-heptene, 1-octene,  
2,3-dimethyl-1-butene, 2,3-dimethyl-2-butene), cycloalkenes (cyclopentene,  
cyclohexene), cycloalkanes (cyclopentane, cyclohexane, methylcyclopentane,

cycloheptane, cyclooctane), aroms. and their relatives (benzene, ethylbenzene, o-, m-, and p-xylenes, propylbenzene, isopropylbenzene, hemimellitene, pseudocumene, mesitylene, 1,3-cyclohexadiene, 1,4-cyclohexadiene, bicyclo[2.2.1]hepta-2,5-diene [norbornadiene]), and deuterated hydrocarbons (benzene-d<sub>6</sub>, cyclohexane-d<sub>12</sub>, toluene-d<sub>8</sub>). Measurements were made with the transient hot-wire method in the manner previously presented, and the thermal diffusivity values were corrected by reference to the heat capacity of heptane as a reference material. Heat capacities (volume, c<sub>pp</sub>; mass, c<sub>p</sub>; molar, C<sub>m,p</sub>) were complementally derived from the relationship  $c_{pp} = \lambda/a$ , with values for the d. and the molar mass. The uncertainty of the data is estimated to be 0.4 % for the thermal conductivity (absolutely measured) and about 1.8 % for the thermal diffusivity (with a coverage factor of k<sub>p</sub> = 2; p = 95%), although that of  $\lambda$  (not of a) is possibly inferior for a few substances containing slightly more impurities (i.e., mainly other isomers).

REFERENCE COUNT: 60 THERE ARE 60 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 3 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2003:925818 CAPLUS

DOCUMENT NUMBER: 140:184519

TITLE: Green diesel from biomass via Fischer-Tropsch synthesis: new insights in gas cleaning and process design

AUTHOR(S): Boerrigter, Harold; Uil, Herman den; Calis, Hans-Peter  
CORPORATE SOURCE: Unit ECN Biomass, Energy research Centre of the Netherlands (ECN), Petten, 1755 ZG, Neth.

SOURCE: Pyrolysis and Gasification of Biomass and Waste, Proceedings of an Expert Meeting, Strasbourg, France, Sept. 30-Oct. 1, 2002 (2003), Meeting Date 2002, 371-383. Editor(s): Bridgwater, A. V. CPL Press: Newbury, UK.

CODEN: 69EUGL; ISBN: 1-872691-77-3

DOCUMENT TYPE: Conference

LANGUAGE: English

AB The interest in biomass is increasing in the light of the growing concern about global warming and the resulting climate change. The emission of the greenhouse gas CO<sub>2</sub> can be reduced when green biomass-derived transportation fuels are used. One of the most promising routes to produce green fuels is the combination of biomass gasification (BG) and Fischer-Tropsch (FT) synthesis, wherein biomass is gasified and after cleaning the biosyngas is used for FT synthesis to produce long-chain hydrocarbons that are converted into green diesel. To demonstrate this route, a small FT unit based on Shell technol. was operated for in total 650 h on biosyngas produced by gasification of willow. In the studied system, tars were removed in a high-temperature tar cracker and other impurities, like NH<sub>3</sub> and H<sub>2</sub>S were removed via wet scrubbing followed by active-carbon and ZnO filters. The exptl. work and the supporting system anal. afforded important new insights on the desired gas cleaning and the optimal line-up for biomass gasification processes with a maximized conversion to FT liqs. Two approaches were considered: a front-end approach with reference to the (small) scale of existing circulating fluidized bed gasifiers (1-100 MWth) and a back-end approach with reference to the desired (large) scale for FT synthesis (500-1000 MWth). In general, the sum of H<sub>2</sub> and CO in the raw biosyngas is an important parameter, whereas the H<sub>2</sub>/CO ratio is less relevant. BTX (i.e. benzene, toluene, and xylenes) are the design guideline for the gas cleaning and with this the tar issue is de-facto solved (as tars are easier to remove than BTX). To achieve high yields of FT products the presence of a tar cracker in the system is required. Oxygen gasification allows a further increase in yield of FT products as a N<sub>2</sub>-free gas is required for off-gas recycling. The scale of the BG-FT installation detts. the line-up of the gas cleaning and the integrated process. It is

expected that the future of BG-FT systems will be large plants with pressurized oxygen blown gasifiers and maximized Fischer-Tropsch synthesis.

REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 4 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2001:333844 CAPLUS

DOCUMENT NUMBER: 135:7411

TITLE: Recovery of energy from gaseous effluents

AUTHOR(S): Anon.

CORPORATE SOURCE: UK

SOURCE: Research Disclosure (2001), 444(April), P599 (No. 444108)

CODEN: RSDSBB; ISSN: 0374-4353

PUBLISHER: Kenneth Mason Publications Ltd.

DOCUMENT TYPE: Journal; Patent

LANGUAGE: English

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
RD 444108		20010410		

PRIORITY APPLN. INFO.: RD 2001-444108 20010410

AB During an exothermic gas-liquid reaction, e.g. oxidation and hydrogenation, normally a large amount of reaction enthalpy has to be removed from the reaction mixture. This can be accomplished by direct cooling of the liquid reaction mixture over an ordinary heat exchanger, but it is also possible to remove the reaction enthalpy by evaporation. Typically, the off gas of such a reactor consists of the same components as the liquid phase, inert gasses from the gas feed and possible non-condensable byproducts. The concentration

of

the various components in the gas phase is determined by the composition of the liquid

phase and the vapor-liquid equilibrium of the various components, as can be described by Raoult's law. In order to reduce the loss of energy, and thus improving the overall process efficiency, it is common practice to recover the energy from the off gas stream by direct or indirect exchanging of heat with a coolant. During this process, possibly complete or partial condensation takes place. In order to cool the off gas several technol. solns. can be employed, e.g. cooling the off gas in a heat exchanger and cooling the off gas by direct contact with a cooling medium. When an indirect heat exchanger is applied, the possibility to recover the heat (e.g. in the production of steam) depends strongly on the actual temperature of

the off gas. At lower temps., most of the present energy cannot be recovered in a useful form because of the second law of thermodyn. and has to be purged to the environment. In alternative solns., it is also possible to heat up an arbitrary process flow instead of a coolant. However, it is also possible to use a direct heat exchanger, in which a cold liquid is in direct contact with the off gas containing the volatile components (condensibles), stripped from the reactor. This will result in a cooled off gas and heated liquid stream. The cold liquid may consist of anything, but in an elegant set up, the liquid is the same as the starting material, that is fed to the reactor. In that case, the volatile components will try to attain their vapor-liquid equilibrium (as described by Raoult's law) and will be transported to the liquid phase. When a counter current flow is employed in this heat exchanger, a number of vapor-liquid equilibrium stages can be created, in which it is possible to remove impurities from the off gas almost quantitatively. The vapor-liquid equilibrium of all condensable components will cause a concentration of the highest boiling components in the liquid phase. In case the product components have a higher b.p. than the starting material, this method also allows to increase the recovery yield of the product, if the liquid effluent is transferred directly into the product recovery section. The concentration of

these components in the original off gas will be increased by preferential condensation because of the b.p. differences. The gaseous effluent may be further cooled down to condense and remove the last traces of organic substances before release into the environment. The above mentioned concept may be useful in the hydrogenation of organic compds., like carboxylic acids (e.g. phthalic acid and fumaric acid), aldehydes and alcs., and in the oxidation of organic compds. such as cyclohexene, styrene, butene, benzene and alkyl substituted benzene's like cumene, toluene and xylenes.

L12 ANSWER 5 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1994:703532 CAPLUS  
DOCUMENT NUMBER: 121:303532  
TITLE: Nonextractive process for producing high-purity aromatics from a feedstock containing nonaromatic impurities  
INVENTOR(S): Innes, Robert A.; Mulaskey, Bernard F.; Wall, Robert G.; Shebek, Peter M.  
PATENT ASSIGNEE(S): Chevron Research and Technology Co., USA  
SOURCE: PCT Int. Appl., 27 pp.  
CODEN: PIXXD2  
DOCUMENT TYPE: Patent  
LANGUAGE: English  
FAMILY ACC. NUM. COUNT: 2  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9400409	A1	19940106	WO 1993-US5814	19930616
W: AT, AU, BB, BG, BR, CA, CH, CZ, DE, DK, ES, FI, GB, HU, JP, KP, KR, KZ, LK, LU, MG, MN, MW, NL, NO, NZ, PL, PT, RO, RU, SD, SE, SK, UA, VN				
RW: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
AU 9345390	A	19940124	AU 1993-45390	19930616
DE 4393010	C1	19970507	DE 1993-4393010	19930616
ZA 9304391	A	19940117	ZA 1993-4391	19930618
CN 1098084	A	19950201	CN 1993-107986	19930724
GB 2279661	A	19950111	GB 1994-16348	19940812
GB 2279661	B	19960501		

PRIORITY APPLN. INFO.: US 1992-900857 A 19920618  
WO 1993-US5814 A 19930616

AB The process comprises contacting a feedstock containing  $\geq 70\%$  aroms. and  $\geq 0.5\%$  close-boiling paraffin and naphthene nonaroms. with an acidic catalyst to convert the close-boiling nonaroms. to lower and higher boiling hydrocarbons, and recovering a liquid aromatic product by distillation, wherein the product contains  $\geq 99\%$  aroms. A feedstock containing 92:8 PhMe and C7 nonaroms. was reacted over an acidic  $\beta$ -zeolite catalyst at  $316^\circ$  and 600 psig to give disproportionated products containing benzene and xylenes while removing close-boiling nonaroms. by cracking.

L12 ANSWER 6 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1994:419113 CAPLUS  
DOCUMENT NUMBER: 121:19113  
TITLE: Synthesis and characterization of SAPO-5 molecular sieve using N,N-dimethylbenzylamine as template  
AUTHOR(S): Prakash, A. M.; Unnikrishnan, S.; Rao, K. V.  
CORPORATE SOURCE: Dep. Phys., Sardar Patel Univ., Vallabh Vidyanagar, 388 120, India  
SOURCE: Microporous Materials (1994), 2(2), 83-9  
CODEN: MCMTEV; ISSN: 0927-6513  
DOCUMENT TYPE: Journal  
LANGUAGE: English  
AB The synthesis of SAPO-5 mol. sieves with high silicon contents has been

attempted using N,N-dimethylbenzylamine as templating agent. SAPO-5 samples having silicon contents up to 35%, relative to total Si+Al+P, have been achieved. An increase in concentration of the templating agent in the synthesis gel effects increased silicon substitution albeit the formation of certain impurity phases. Characterization of such silicon-rich SAPO-5 materials has been carried out by various physiochem. techniques such as XRD, SEM, EDAX, MAS NMR, IR, TGA/DTA, TPD and a catalytic reaction, viz. the alkylation of toluene with methanol to xylenes.

L12 ANSWER 7 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1989:576685 CAPLUS

DOCUMENT NUMBER: 111:176685

TITLE: Isomerization of m-xylene and ethylbenzene on zeolite-containing catalysts

AUTHOR(S): Komarov, V. S.; Bolotnikova, E. V.; Shirinskaya, L. P.

CORPORATE SOURCE: Inst. Obshch. Neorg. Khim., Minsk, USSR

SOURCE: Vestsi Akademii Navuk BSSR, Seryya Khimichnykh Navuk (1989), (3), 3-6

CODEN: VBSKAK; ISSN: 0002-3590

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB The activity of aluminosilicate catalysts in m-xylene (I) isomerization was enhanced by introduction of 5-80% decationized superhigh-silica zeolites of the TsVM or TsVK type into an aluminosilicate matrix. The activity increased with increasing zeolite content. An aluminosilicate matrix with 30% Al<sub>2</sub>O<sub>3</sub> content provided zeolite-containing catalysts for I isomerization at high activity and selectivity, especially at elevated temps. (350-400°). The formation of o- and p-xylenes from I isomerization increased on increasing the temperature from 250 to 400°, but at 380-400° isomerization was accompanied by disproportionation, resulting in the formation of toluene, benzene, and trimethylbenzenes. Disproportionation was also the main pathway of isomerization of ethylbenzene impurity in I, with the main products being benzene, toluene, diethylbenzenes, and triethylbenzenes.

L12 ANSWER 8 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1983:209383 CAPLUS

DOCUMENT NUMBER: 98:209383

TITLE: Gas-chromatographic analysis of trace impurities for ethylbenzene, o-, m- and p-xylenes in toluene

AUTHOR(S): Kugucheva, E. E.; Puchkova, N. A.; Printsman, E. A.

CORPORATE SOURCE: VNIIOS, Moscow, USSR

SOURCE: Neftepererabotka i Neftekhimiya (Moscow, Russian Federation) (1983), (3), 35-6

CODEN: NNNSAF; ISSN: 0028-1190

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB The total xylene content and individual xylene isomer contents, in particular p-xylene, in toluene used as raw material for the liquid-phase oxidation production of benzoic acid were determine by

a gas chromatog. method (min. sensitivity to xylene impurity 3-5 ppm. by weight). The chromatog. LKhM 8MD (Model 5 with flame ionization detector in the 10-12 A sensitivity range) was used with a 4-m long column packed with 25% p,p'-azoxyphenetole on Chromosorb P at the following conditions: thermostat temperature 140°; vaporizer temperature 250°; gas carrier 10 mL N/min; and sample size 0.5 µL. With this liquid crystal packing, p-xylene emerged second, not first, and with less overlap by the toluene peak. Mixed phase packings, such as Benton-245 with PFMS-4 or Benton-34 with diisodecyl phthalate were not useful because of the toluene peak overlap of the impurity peaks.

XYLENE IS AN  
IMPURITY IN  
TOLUENE

L12 ANSWER 9 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1980:198017 CAPLUS

DOCUMENT NUMBER: 92:198017

TITLE: Technology for industrial preparation of crude benzene for further processing

AUTHOR(S): Grabiec-Koska, Wanda; Przybylo, Kazimierz; Miller, Grazyna

CORPORATE SOURCE: Biuro Projekt. Realizacji Inwest "Prosynchem", Gliwice, Pol.

SOURCE: Koks, Smola, Gaz (1979), 24(9), 259-64

CODEN: KSMGAA; ISSN: 0023-2823

DOCUMENT TYPE: Journal

LANGUAGE: Polish

AB Initial processing of cokery-supplied crude benzene is described. The crude product contains benzene 65-75, toluene 10-15, and xylenes 2-4 weight% as well as water and suspended solid coal, coke, mineral matter, and rust particles. The impurities all separated as an aqueous sludge by 30 day sedimentation and 3-stage filtration of the feedstock prior to distillation. The benzene-containing waste waters are processed sep.

L12 ANSWER 10 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1980:58316 CAPLUS

DOCUMENT NUMBER: 92:58316

TITLE: Isolation of aromatic hydrocarbons

AUTHOR(S): Grishchenko, N. F.; Yablochkina, M. N.; Shapiro, L. P.; Rogozkin, V. A.

CORPORATE SOURCE: USSR

SOURCE: Khimiya i Tekhnologiya Topliv i Masel (1979), (10), 26-9

CODEN: KTPMAG; ISSN: 0023-1169

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB Reforming narrow ligroine fractions (b. at 62-85, 62-105 and 105-40°) gave mixts. of aromatic hydrocarbons, from which high-purity C<sub>6</sub>H<sub>6</sub>, PhMe and xylene were isolated by extraction with HO(CH<sub>2</sub>CH<sub>2</sub>O)<sub>4</sub>H containing 5% H<sub>2</sub>O and then selective hydrogenation over 0.1-0.15% Pt/Al<sub>2</sub>O<sub>3</sub> to remove unsatd. impurities.

L12 ANSWER 11 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1978:114871 CAPLUS

DOCUMENT NUMBER: 88:114871

TITLE: Determination of trace impurities in toluene by gas chromatography-chemical ionization mass spectrometry

AUTHOR(S): Iida, Yoshio; Okada, Shizuko

CORPORATE SOURCE: Fac. Eng., Seikei Univ., Tokyo, Japan

SOURCE: Bunseki Kagaku (1977), 26(9), 630-4

CODEN: BNSKAK; ISSN: 0525-1931

DOCUMENT TYPE: Journal

LANGUAGE: Japanese

AB 2-Methylbutane, CS<sub>2</sub>, cyclopentane, 2-methylpentane, 3-methylpentane, n-hexane, methylcyclopentane, C<sub>6</sub>H<sub>6</sub>, cyclohexane, n-heptane, methylcyclohexane, 3-methylthiophene, PhEt, and xylenes were determined in reagent-grade PhMe by using gas chromatog. and chemical-ionization mass spectroscopy. The anal. conditions were: stationary phases, 5% polyethylene glycol or 5% squalane; column temps. 50 or 65°; carrier gas, CH<sub>4</sub>; ion source temperature, 80°; and ion energy, 120 eV. The determination limits were 10-50 pg. impurity.

L12 ANSWER 12 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1975:595764 CAPLUS



DOCUMENT NUMBER: 83:195764  
TITLE: Fractionation column control  
AUTHOR(S): Boyd, D. M.  
CORPORATE SOURCE: Process Div., Univers. Oil Prod. Co., Des Plaines, IL, USA  
SOURCE: Chemical Engineering Progress (1975), 71(6), 55-60  
CODEN: CEPRA8; ISSN: 0360-7275

DOCUMENT TYPE: Journal  
LANGUAGE: English

AB The composition profile in the column is used for control. Temperature differences

between 2 trays above the feed tray and 2 trays below are measured. The 2 differential temperature signals are fed to a subtraction relay. The difference

between these signals is the control signal. The method was tested on the distillation of a solvent-extracted aromatic hydrocarbon mixture with C<sub>6</sub>H<sub>6</sub> being the

overhead product and the bottom product containing PhMe (50%), xylenes, and PhEt. The column had 70 trays. The overhead product was taken off tray 5 and feed was on tray 39. The differential temperature measurements were made between theor. trays 5 and 15 and 30 and 45. Calcns. showed that PhMe 10 ppm in the overhead would produce a control signal of +10°F while C<sub>6</sub>H<sub>6</sub> 10 ppm in the bottoms would produce a signal of -15°F. Changing the overhead product from 100,000 to 100,030 moles would change the differential temperature signal from -30° to +10°F. However, feedforward control was shown to be unnecessary and the controllers were stable at a proportional band setting of 20% with a reset rate of 5 min and a derivative of 1 min. The control method was used successfully on C<sub>6</sub>H<sub>6</sub>-PhMe columns connected in series in a refinery. The field showed that, with computer calcns. and measurement of the double differential temperature in the column, the impurities can be predicted more closely than the chromatographs available in the average plant control

laboratory  
can determine

L12 ANSWER 13 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1973:494570 CAPLUS

DOCUMENT NUMBER: 79:94570

TITLE: Distribution of pure benzene impurities  
along the height of a fractionation column

AUTHOR(S): Nabivach, V. M.; Gulyaev, Yu. I.; Yatsenko, S. S.

CORPORATE SOURCE: USSR

SOURCE: Metallurgiya i Koksokhimiya (1972), No. 32, 78-80

CODEN: MEKOAG; ISSN: 0543-5765

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB The distribution impurities in C<sub>6</sub>H<sub>6</sub> and its variation along the height of a fractionation column during 10 months' production were studied by using gas chromatog. The total content of impurities varied in the range 0.25-0.50%. The C<sub>6</sub>H<sub>6</sub> content in the sample increased with

temperature

during transition to higher plates of the column. Analogous variations were found for n-C<sub>7</sub>H<sub>16</sub>, methylcyclopentane, and cyclohexane.

Impurities with higher b.ps. (toluene and xylenes) varied in an inverse manner.

L12 ANSWER 14 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1973:421438 CAPLUS

DOCUMENT NUMBER: 79:21438

TITLE: Acid free process for isolating mesitylene from crude benzene

AUTHOR(S): Kolyandr, L. Ya.; Privalov, V. E.; Tyaptina, M. I.; Gapotchenko, N. P.; Kochergin, V. A.; Khvatkov, N. M.; Krishtopa, A. P.; Bilym, L. M.

CORPORATE SOURCE: Ukr. Nauchno-Issled. Uglekhim. Inst., Kharkov, USSR

SOURCE: Koks i Khimiya (1973), (4), 29-34  
CODEN: KOKKAI; ISSN: 0023-2815

DOCUMENT TYPE: Journal

LANGUAGE: Russian

AB Mesitylene and o-ethyltoluene can not be separated by rectification. In the process developed, o-ethyltoluene was removed by disproportionation and isomerization, through heating at 75-p°, with 7% AlCl<sub>3</sub> for 120-140 min. After hydrolysis of AlCl<sub>3</sub> and settling, the mesitylene was separated from m- and p-ethyltoluene, pseudocumene, toluene, and xylenes, by rectification at 500-o mm Hg to give a product containing 98.4-98.7% pure mesitylene, the impurities being saturated hydrocarbons.

L12 ANSWER 15 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1972:437351 CAPLUS

DOCUMENT NUMBER: 77:37351

TITLE: Catalytic hydrogenation of aromatic hydrocarbons to naphthenes

INVENTOR(S): Cessou, Maurice; Cosyns, Jean

PATENT ASSIGNEE(S): Institut Francais du Petrole, des Carburants et Lubrifiants

SOURCE: Ger. Offen., 12 pp.

CODEN: GWXXBX

DOCUMENT TYPE: Patent

LANGUAGE: German

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2142917	A	19720309	DE 1971-2142917	19710827
DE 2142917	B2	19810312		
DE 2142917	C3	19820107		
FR 2105481	A5	19720428	FR 1970-32661	19700908
US 3798279	A	19740319	US 1971-176320	19710830
NL 7111950	A	19720310	NL 1971-11950	19710831
NL 171151	B	19820916		
NL 171151	C	19830216		
BE 772207	A1	19720306	BE 1971-3363	19710906
GB 1313084	A	19730411	GB 1971-41496	19710906
JP 54003866	B	19790227	JP 1971-68837	19710906

PRIORITY APPLN. INFO.: FR 1970-32661 A 19700908

AB Naphthenes containing <100 ppm impurities were prepared by 2-stage hydrogenation of aromatic compds. using a Ni or Co Raney catalyst and a Ni or Co oxide-supported catalyst in the 2nd stage. Thus, C<sub>6</sub>H<sub>6</sub> containing C<sub>6</sub> 30, C<sub>7</sub> 250, cyclohexane 100, and PhMe 20 ppm was hydrogenated on Raney Ni at 200° and 40 kg/cm<sup>2</sup> and then catalytically hydrogenated at 180-240° and 6 kg/cm<sup>2</sup>. The catalyst was prepared by impregnating Al<sub>2</sub>O<sub>3</sub> (surface area 250 m<sup>2</sup>/g and porosity 0.65 cm<sup>3</sup>/g) with NiNO<sub>3</sub>, calcining at 300°, and hydrogenating at 350° and 2 kg/cm<sup>2</sup>. Chemisorption of 0.25 cm<sup>3</sup> H/g catalyst took place. The impurity content of the cyclohexane was <100 ppm. A catalyst hydrogenated at 350° having a chemisorption of 2.5 cm<sup>3</sup> H/g gave naphthenes containing >400 ppm impurities. The Co catalyst operated at a lower temperature. Production of methylcyclohexane from toluene and of dimethylcyclohexanes from xylenes is similarly accomplished.

L12 ANSWER 16 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1971:88997 CAPLUS

DOCUMENT NUMBER: 74:88997

TITLE: Use of process gas chromatographs in installation for the production of aromatic hydrocarbons

AUTHOR(S): Terada, Osamu

CORPORATE SOURCE: Nihon Pet. Chem. Co. Ltd., Japan

SOURCE: Aromatikkuusu (1971), 23(1), 17-23

CODEN: AROMBO; ISSN: 0365-6187

DOCUMENT TYPE: Journal  
LANGUAGE: Japanese

AB Impurities in C6H6, PhMe, and xylene products were analyzed with process gas chromatographs. Nonaromatics (isoparaffins and paraffins) 200 and PhMe 500 ppm in C6H6 could be determined Nonaromatics 500, C6H6 5000, and C8 aromatics 5000 ppm in PhMe could be determined Nonaromatics 500, PhMe 5000, and C9 aromatics 5000 ppm in xylenes could be determined Hardware, software, etc. are discussed.

L12 ANSWER 17 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1970:81266 CAPLUS  
DOCUMENT NUMBER: 72:81266  
TITLE: Purity of by-product coke toluene  
AUTHOR(S): Nabivach, V. M.; Yatsenko, S. S.  
CORPORATE SOURCE: Dnepropetrovsk. Khim.-Tekhnol. Inst. im.

Dzerzhinskogo, Dnepropetrovsk, USSR  
SOURCE: Zhurnal Prikladnoi Khimii (Sankt-Peterburg, Russian Federation) (1969), 42(12), 2797-802  
CODEN: ZPKHAB; ISSN: 0044-4618

DOCUMENT TYPE: Journal  
LANGUAGE: Russian

AB Quant. gas-chromatog. of aliphatic, alicyclic, and aromatic hydrocarbons and thiophenes in PhMe was studied. Separation took place at 95° on a composite column: the 1st section (2 m long) was filled with Chromosorb W + 18% Rheoplex 400; the 2nd section (3 m long) was filled with Chromosorb W + 18% Tween 20. The method was applied to pure coke oven PhMe from various plants. Methylcyclohexane, n-C8H18, 1,2-trans-dimethylcyclohexane, C6H6, n-C9H20, cyclooctane, PhEt, and p- and m-xylenes were identified and determined quant. The purity of the PhMe was 98.45-98.96%.

L12 ANSWER 18 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1970:81182 CAPLUS  
DOCUMENT NUMBER: 72:81182  
TITLE: Separation of aromatic from nonaromatic hydrocarbons  
INVENTOR(S): Anderson, John R.; Somekh, George S.  
PATENT ASSIGNEE(S): Union Carbide Corp.  
SOURCE: U.S., 7 pp.

CODEN: USXXAM  
DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3492365	A	19700127	US 1966-553336	19660527
BE 741522	A	19700511	BE 1969-741522	19691110
			US 1966-553336	A 19660527

PRIORITY APPLN. INFO.:

AB Benzene, toluene, and xylenes were separated from a mixed hydrocarbon feed by extraction with solvents and distillation of the extract Thus, the

feed was introduced into the midpoint of a primary extraction column. Dry triethylene glycol was introduced at the top of the column and areflux containing (volume %) 39.9% secondary solvent (normal paraffins b. 175-248°), 9.0% benzene, 21.0% toluene, and 30.0% xylenes was fed at the bottom. The primary extract from this stage was (volume %) 4.8% benzene, 6.5% toluene, 6.6% xylenes, 1.2% secondary solvent, and 80.9% triethylene glycol. Because no feed components other than benzene, toluene, and xylenes were in the extract, a final disn. completed the separation Changing operating parametes in the primary column gave some nonaromatic feed impurities in the primary extract It was subjected to a secondary extraction in the same column with secondary solvent introduced at the bottom.

Hydrocarbon-free triethylene glycol was the bottoms product and secondary extract the overhead. The secondary extract was distilled at 1 atm in a batch still. The distillate was recovered in 3 fractions as follows (volume % purity given): benzene 99, toluene 99+, and xylenes 99. The process is also operable in a continuous manner.

L12 ANSWER 19 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1965:82151 CAPLUS  
DOCUMENT NUMBER: 62:82151  
ORIGINAL REFERENCE NO.: 62:14531f-g  
TITLE: Chromatography of aromatic isomers in the products of p-xylene manufacture  
AUTHOR(S): Vigdergauz, M. S.; Chabrova, O. G.  
CORPORATE SOURCE: Res. Inst. Synthetic Alcohols and Organic Products, Novokuibyshevsk  
SOURCE: Neftekhimiya (1965), 5(1), 160-5  
CODEN: NEFTAH; ISSN: 0028-2421  
DOCUMENT TYPE: Journal  
LANGUAGE: Russian

AB The products of catalytic conversion of petroleum hydrocarbons into p-xylene (I) were identified by gas chromatography at 75° in a 1.2-m. long column. The stationary phase was Chromosorb W (II) containing 20% of a mixture of Bentone-34 (III) and dinonyl phthalate (60:40). This chromatographic system was also used for the separation of mixts. of propylbenzene, cumene (IV), and isomeric ethylmethylbenzenes. Impurities in com. IV and products of pyrolysis of IV were determined by chromatography on a mixture of III and silicone oil (1:1). Mixts. of C8 aromatic hydrocarbons were separated on a stationary phase consisting of II containing 15% of a mixture of Bentone-245 and vaseline oil (28:72) (CA 62, 7551b).

L12 ANSWER 20 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1964:418052 CAPLUS  
DOCUMENT NUMBER: 61:18052  
ORIGINAL REFERENCE NO.: 61:3032g-h,3033a-b  
TITLE: Production of aromatic nitriles  
INVENTOR(S): Kroeper, Hugo; Platz, Rolf; Nohe, Heinz; Schanz, Rudi  
PATENT ASSIGNEE(S): Badische Anilin- & Soda-Fabrik AG  
SOURCE: 20 pp.  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BE 632702		19631122	BE	
DE 1172253			DE	
FR 1357429			FR	
GB 972122			GB	
US 3278573		19661011	US 1963-281761	19630520
PRIORITY APPLN. INFO.:			DE	19620523

AB A process was described for the production of aromatic nitriles and dinitriles by oxidation of aromatic hydrocarbons with O and NH3 over V2O5-CrO3 catalysts in Al2O3 carriers. A complete method for the preparation and the control of the properties of several catalysts with V2O5-CrO3 ratios varying from 10:1 to 1:10, and over several precipitated aluminas (v, δ, η, .vkappa., or .vtheta. type) carriers, is described. NH3 (75 l./hr.), N (150 l./hr.), and air (75 l./hr.) are passed through the catalyst (200 cc.), placed in a vertical reaction tube and heated at between 380° and 460°. The hydrocarbon (toluene, ethylbenzene, xylenes, alkylbenzenes, mesitylene, 1-methylnaphthalene, 1,8-diethylnaphthalene, or 3,4-dimethylbiphenyl) is vaporized, and introduced (0.5-3%) into the preceding gas stream, being the reaction time from 0.1 to 25 sec. (preferably 0.2-5 sec.). The

reaction product condensed, and washed with water gives benzonitrile, phthalonitriles, trimesonitrile, 1-naphthonitrile, 1,8-naphthodinitrile, or 4-phenylphthalonitrile. Tables are given with the catalysts, reaction times, yields, and % of impurities. The best conditions to obtain o-phthalonitrile (65% with less than 0.5% of phthalimide, and phthalamide) are: a gas stream with 2.7% o-xylene, 10% O, and 87.3% NH<sub>3</sub>, a reaction time of 0.5 sec., a reaction temperature of 400°, and a suitable catalyst with an  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> carrier.

L12 ANSWER 21 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:477937 CAPLUS  
DOCUMENT NUMBER: 59:77937  
ORIGINAL REFERENCE NO.: 59:14550f-h, 14551a  
TITLE: Use of certain organic substances as polarographic supporting electrolytes  
AUTHOR(S): Malinina, R. D.  
SOURCE: Sb. Tr. Tsent. Nauchn.-Issled. Inst. Chernoi Met. (1963), (31;Pt. 1), 158-61  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

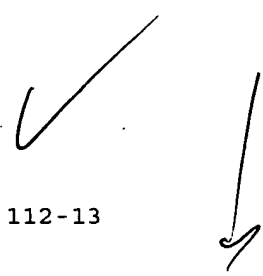
AB Tartaric acid, Trilon B, ethylenediamine (I), and dimethyl-p-phenylenediamine (II) solns. were tested as possible polarographic supporting electrolytes for the determination of impurities in the presence of interfering elements in standard solns. of Bi, Sb, Pb, Cd, Ni, W, Co, Te, Cu, Zn, Sn, and Mo (25 ml. of 0.005% solution of each of the above elements). In 0.1M tartaric acid (pH 3), Bi, Sb, Pb, Cd, Cu, Te, Zn, and Mo are reduced on a dropping Hg electrode: a very good wave is obtained for Te. Mo gives 2 waves; at pH = 8, Mo is not reduced, but the reduction potentials of the remaining elements are displaced toward more neg. values; Pb at pH = 8 in 0.1M tartaric acid does not give a wave. In 0.3M I at pH 3, good polarograms are obtained for Sb, Te, and Mo, worse polarograms are obtained in the reduction of Pb, W, and Cu, and the remaining elements are not reduced; at pH = 8, reduction of Ni, Co, Cd, Zn, and Mo is observed. With II at pH 3, reduction of Bi, Pb, Sb, Cd, Cu, and Te is observed, and the remaining elements are not reduced. Good polarograms are obtained for Te, Cu, Cd, and Sb in 0.035M Trilon B at pH 3; for Mo, 2 well-expressed waves are observed; reduction of Ni, W, Co, Zn, and Sn is not observed. In 0.1M tartaric acid and 0.3M I solution at pH 3 reduction of Bi,

Sb, Pb, Cd, Te, Cu, and Mo is observed; Ni, W, Co, Zn, and Sn do not give any wave; increase of pH to 8 displaces the reduction potentials of these elements. In 0.1M tartaric acid and 0.035M Trilon B solution at pH 3, only Bi, Sb, and Cu are reduced. Almost the same results are obtained in a solution containing a mixture of 0.3M I and 0.035M Trilon B, except for Te

which gives a very small wave. In a mixture of 0.3M I and 0.1M II at pH = 3, well-expressed polarograms of Sb and Cd are obtained; in this solution, reduction of Te, Cu, and Mo is observed. II solution is recommended as a supporting electrolyte in the determination of microquantities of Sb (0.002-0.005%), and the mixture of 0.3M I and 0.035M Trilon B for the polarography of Bi, Sb, and Cu in the presence of Pb, Cd, Ni, Co, W, Zn, and Sn.

L12 ANSWER 22 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:477936 CAPLUS  
DOCUMENT NUMBER: 59:77936  
ORIGINAL REFERENCE NO.: 59:14550f  
TITLE: Impurities in analytical reagent grade chemicals  
AUTHOR(S): Hogfeldt, E.  
CORPORATE SOURCE: Roy. Inst. Technol., Stockholm  
SOURCE: Journal of Chromatography (1963), 12(1), 112-13  
CODEN: JOCRAM; ISSN: 0021-9673  
DOCUMENT TYPE: Journal



LANGUAGE: Unavailable

AB Gas chromatography is useful for detecting impurities in so-called anal. reagents, particularly volatile organic compds., and is advocated for routine checks by manufacturers of reagents. An example of impurities in xylene is given.

L12 ANSWER 23 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:468788 CAPLUS

DOCUMENT NUMBER: 59:68788

ORIGINAL REFERENCE NO.: 59:12662b-e

TITLE: Catalytic process for the purification of light hydrocarbons by liquid-phase hydrogenation

INVENTOR(S): Limido, Jean; Miguel, J.; Thonon, C.

PATENT ASSIGNEE(S): Institut Francais du Petrole, des Carburants et Lubrifiants

SOURCE: Addn. to Fr. 1,237,708 (CA 55, 16985a)

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 73914		19610424	FR	

AB Methods of selective hydrogenation of impurities in light hydrocarbons based on the use of a heavy paraffinic solvent and a Group VIII metal catalyst are described. These methods are recommended for feeds of hydrocarbons with <16 C atoms. When impurities such as S are present, the process is operated in 2 stages: hydrotreating of a paraffinic solution using an oxide or sulfide catalyst, followed by hydrogenation with a metal catalyst. The product can be obtained by distillation C6H6 in paraffin oil (1:3) at 195°/12 atmospheric with Raney Ni-H gave 100% cyclohexane in 15 min. Reduction of equal vols. of xylenes and dodecylcyclohexane (as solvent) at 150°/8 atmospheric with Ni on diatomaceous earth gave 100% dimethylcyclohexane in 30 min. Equal vols. of isooctene and paraffin oil (as solvent) at 195°/3 atmospheric with H and Ni on diatomaceous earth gave 90% isooctane in 1 hr. Equal vols. of toluene and cetane (as solvent) at 130°/15 atmospheric with H and Pt on active C gave 100% methylcyclohexane in 1 hr. Hydrogenation of cyclohexane of dicyclohexyl (1:2) at 200°/15 atmospheric with Ni on alumina gave 100% cyclohexane in 35 min. A hydrocarbon fraction, b. 150-200°, 77.5% saturated, and 22.5% aromatic hydrocarbons containing 0.07% S, was treated in 2 stages. First, a solution of equal vols. of the fraction and a gas oil solvent at 375°/40 atmospheric with a mixture of Mo and Co oxides deposited on alumina as a catalyst was treated with H for 40 min. On distillation, a fraction containing 78.5% saturated and 21.5% aromatic hydrocarbons and 0.001% S was obtained. This was dissolved in an equal volume of gas oil and treated with H at 200°/20 atmospheric with Ni on diatomaceous earth as catalyst for 80 min. On distillation a light fraction containing only 0.9% aromatics was obtained.

L12 ANSWER 24 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:408755 CAPLUS

DOCUMENT NUMBER: 59:8755

ORIGINAL REFERENCE NO.: 59:1546c-d

TITLE: Sulfonation of aromatic hydrocarbons with dioxane-sulfur trioxide complex

AUTHOR(S): Gordash, Yu. T.; Laryutina, E. A.; Semyachko, R. Ya.

SOURCE: Doklady Akademii Nauk BSSR (1962), 6, 237-9

CODEN: DBLRAC; ISSN: 0002-354X

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB Sulfonation of PhMe, m- and p-xylenes, and mesitylene in C2H4Cl2 with dioxane-SO3 complex 2 hrs. at 20° and neutralization with NaOH

gave nearly quant. yields of Na sulfonates. With o-xylene and  $\alpha$ - and  $\beta$ -methylnaphthalenes, petr. ether exts. of the neutralized product solns. gave 30, 24 (m.  $94^\circ$ ,  $C_{12}H_{14}SO_2$ ), and 7.5%, resp., white crystalline solids, believed present in starting materials. With correction for impurities, yields of the latter 3 sulfonates are near quant. No tar formation was observed.

L12 ANSWER 25 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:32468 CAPLUS

DOCUMENT NUMBER: 58:32468

ORIGINAL REFERENCE NO.: 58:5430e-f

TITLE: Determination of nonaromatic hydrocarbons in technically pure aromatics by means of gas chromatography

AUTHOR(S): Sokol., Ludvik

SOURCE: Sb. Praci Vyzkumu Chem. Vyuziti Uhli, Dehtu Ropy (1960), (No. 1), 124-7

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB A gas chromatograph constructed by the author was used for the detns. at  $130^\circ$ . For the analysis of  $C_6H_6$ , 20% triethylene glycol on brick or chromosorb (0.2-0.3 mm. grain size) was used; at higher temps. 15%  $\gamma, \beta$ -dinaphthyl sulfone was used on the same carrier. The column contained 30 g. carrier and its length was 160 cm. The flow of carrier gas (N) was 35-45 ml./min. at an initial pressure of 800-60 mm. Hg. A 0.02-0.1 ml. liquid sample was injected into the column. It is possible to determine  $C_6H_6$  in PhMe, PhMe in xylenes, and vice-versa, and also impurities in  $C_6H_6$  and PhMe in narrow fractions of com. gasoline.

L12 ANSWER 26 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1952:18877 CAPLUS

DOCUMENT NUMBER: 46:18877

ORIGINAL REFERENCE NO.: 46:3246a-d

TITLE: Purification of benzene

INVENTOR(S): Anderson, John R.

PATENT ASSIGNEE(S): Koppers Co., Inc.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2581344		19520108	US 1948-31098	19480604

AB Coke-oven benzene, which contains small amts. of nonaromatic hydrocarbon impurities higher boiling than  $C_6H_6$  and substantially inseparable therefrom by simple distillation, is purified in a 2-stage process which comprises (1) azeotropic distillation of a mixture of the  $C_6H_6$  and an added nonaromatic hydrocarbon material, which boils normally between  $75^\circ$  and  $85^\circ$  and which is present in the mixture in excess of the amount required to form an azeotrope with the  $C_6H_6$ , with the removal of an overhead distillate of  $C_6H_6$  and the added hydrocarbon, and at least a portion of the excess added hydrocarbon and the impurities as a residue; and (2) separation of  $C_6H_6$  and the added azeotrope-forming hydrocarbon by distillation with a polar azeotropic agent in a 2nd still, and recovery of the purified  $C_6H_6$  as a fraction from this still. In the 1st distillation an azeotrope-forming, nonaromatic hydrocarbon is selected which forms with  $C_6H_6$  an azeotrope containing 40-60% of the nonaromatic hydrocarbon. A suitable material is cyclohexane, but any nonaromatic hydrocarbon or any mixture of nonaromatic hydrocarbons b. between  $68^\circ$  and  $90.5^\circ$  can be used effectively in the 1st distillation Aliphatic alcs., such as MeOH or EtOH, and ketones, such as Me<sub>2</sub>CO or MeCOEt, are used effectively in the 2nd distillation They form azeotropes with the cyclohexane or other hydrocarbon

added in the 1st step, and their 2nd-step azeotropes can be readily separated by extraction and decantation with water. For the purification of toluene, ethylbenzene, xylenes, naphthalene, and other aromatic hydrocarbons, the same process is applicable, with a mixture of nonaromatic hydrocarbons derived from petroleum used as the azeotropic agent in the 1st distillation. For example, in the distillation of toluene, a petroleum distillate which contains paraffins and naphthenes and which has a b.p. range from approx. 105° to 112° may be used. The purified toluene may be separated from the azeotrope from the 1st distillation by a 2nd distillation with MeCOEt or MeCN. Cf. C.A. 46, 1235a.

L12 ANSWER 27 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1948:128 CAPLUS  
DOCUMENT NUMBER: 42:128  
ORIGINAL REFERENCE NO.: 42:23a-c  
TITLE: The phase behavior of sodium stearate in anhydrous organic solvents  
AUTHOR(S): Smith, Gerould H.; McBain, James W.  
CORPORATE SOURCE: Stanford Univ., CA  
SOURCE: Journal of Physical and Colloid Chemistry (1947), 51, 1189-1204  
CODEN: JPCCAI; ISSN: 0092-7023  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB Phase diagrams were determined for the systems sodium stearate-hydrocarbon at temps. from 25° to above 310° for the following 12 hydrocarbons: heptane, isooctane, benzene, toluene, ethylbenzene, o-, m-, and p-xylenes, cumene, cyclohexane, p-cymene, and butylbenzene. The soap was soluble to the same extent at any given temperature in all except the last 2 hydrocarbons; this discrepancy was attributed to impurities in these 2 liquids. In every case, the soap and solvent were rigorously dried. All the phase diagrams were similar, exhibiting: (1) a 2-phase gel, (2) a golden or orange liquid-crystallization phase, (3) a white, waxlike liquid-crystallization phase, (4) an isotropic solution, and (5) an isotropic jelly, continuous in phase with the solution. All phase changes were reversible and obeyed the phase rule.

L12 ANSWER 28 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1932:49252 CAPLUS  
DOCUMENT NUMBER: 26:49252  
ORIGINAL REFERENCE NO.: 26:5101b  
TITLE: Hydrocarbons  
PATENT ASSIGNEE(S): Soc. des etablissements Barbet  
DOCUMENT TYPE: Patent  
LANGUAGE: Unavailable  
FAMILY ACC. NUM. COUNT: 1  
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 727095		19320613	FR	19310205

AB Benzene is rectified to obtain C<sub>6</sub>H<sub>6</sub>, toluene and xylenes in 2 sep. operations, the first under atmospheric pressure and the 2nd under reduced pressure. The heat for the 2nd operation is supplied by the vapors from the first. Light impurities are removed in the first and heavier in the 2nd.

L12 ANSWER 29 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1924:19470 CAPLUS  
DOCUMENT NUMBER: 18:19470  
ORIGINAL REFERENCE NO.: 18:2604c-g  
TITLE: Action of sodium sulfite on some trinitro derivatives and on tetranitromethane. Purification of tolite  
AUTHOR(S): Muraour, H.



SOURCE: Bull. soc. chim. [4] (1924), 35, 367-79  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB During the war, because of the shortage of EtOH, use was made of a cold, dilute solution of Na<sub>2</sub>SO<sub>3</sub> for the purification of crude trinitrotoluene (tolite) to produce TNT. The mononitration of C<sub>7</sub>H<sub>8</sub> gives rise principally to a mixture of o- and p-trinitrotoluenes, with small proportion of the m-. On trinitration the o- and p- form the sym. (2,4,6), but the m-passes to both the asym.  $\beta$  (2,3,4) and  $\gamma$  (2,4,5) and sometimes other asym. isomers of these. Moreover, through the oxidizing action of the HNO<sub>3</sub> + H<sub>2</sub>SO<sub>4</sub> mixture at elevated temperature some C(NO<sub>2</sub>)<sub>4</sub> is formed. Also by this

same action TNT gives rise to trinitrobenzoic acid which by the action of the boiling water, used in the purification process, is converted to trinitrobenzene. In addition, there has been observed in the tolite dinitro derivs. of toluene, because the nitration was not completely carried out, and dinitrobenzene and trinitroxylene when the toluene used had not been completely freed from C<sub>6</sub>H<sub>6</sub> and C<sub>8</sub>H<sub>10</sub>. Aqueous solns., containing 3-5% of NaSO<sub>3</sub>, dissolve asym. tolites in the cold, through a sulfo group replacing an NO<sub>2</sub> group in the o-position, and form corresponding Na dinitrosulfonates and NaNO<sub>2</sub>. Methods for the separation of these dinitrosulfonates, in the pure condition, and determination of their constitutions are given. C(NO<sub>4</sub>)<sub>4</sub> is rapidly dissolved, with an elevation in temperature, by a cold sulfite solution containing 4-5% NaSO<sub>2</sub>, giving trinitromethane, NaNO<sub>2</sub> and NaHSO<sub>4</sub>. Eventually, by reaction with the NaNO<sub>2</sub>, there is formed N<sub>2</sub>O<sub>4</sub> and NaC(NO<sub>2</sub>)<sub>3</sub>. The formation of strongly colored, water-soluble, addition compds. with Na sulfite seems to be a general reaction of sym. trinitro derivs. and appears to establish a relation between the sensitiveness of these substances to aqueous NaOH and the readiness of Na<sub>2</sub>SO<sub>3</sub> to dissolve the colored Na salt as it is formed. The introduction of CH<sub>3</sub> groups inhibits this action. The TNT obtained by crystallization from Na<sub>2</sub>SO<sub>3</sub> is not as pure as that obtained from EtOH but is sufficiently so for most of its applications. The variations in the setting points of various samples tested were found due to the different degrees of heterogeneity resulting from the different rates of cooling of the materials. With slow cooling a eutectic rich in the asym. tolites and other impurities is formed. Because of the greater friability of the eutectic, it may be in a large degree separated from the TNT by rubbing through a sieve and this is strikingly demonstrated by exptl. examples. It is concluded that in order to obtain the maximum efficiency in the sulfite treatment, it is essential to use a tolite which has been cooled slowly. Examples are given of ingenious methods, based on the sulfite reaction, for the determination of asym. trinitrotoluenes in crude tolite and for the separation

and determination of mixts. of di- and trinitroxylenes produced in the nitration of crude xylenes. The Pb salts of the oxidation products formed during nitration are very explosive. It is such salts that have covered the Pb coils in EtOH crystallizing vessels and which have given rise to numerous accidents.

L12 ANSWER 30 OF 30 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1921:14573 CAPLUS  
DOCUMENT NUMBER: 15:14573  
ORIGINAL REFERENCE NO.: 15:2708a-g  
TITLE: Standard specification for "benzole" drawn up by the B.E.S.A.  
AUTHOR(S): Anon.  
SOURCE: Automotor J. (1921), 26, p.131  
From: Science Abstracts 24B, 175  
DOCUMENT TYPE: Journal  
LANGUAGE: Unavailable

AB Because of uncertainty as to what constitutes "benzol" or "benzole" used in reference to the motor spirit sold under that name the Brit.

Engineering Standards Associate has issued a standard specification. Definition. The term "benzole" shall denote a liquid consisting essentially of a mixture of benzene, and not more than 30% by volume of toluene and xylenes. Description. The liquid shall be a clear "waterwhite" liquid, free from undissolved water and other visible impurities. Specific gravity. The sp. gr. at 15.5° shall be not less than 0.870, and not more than 0.885. Distillation range. When 100 cu. cm. of the material are distilled in the standard distillation

apparatus

(B.S. Specification, Number 2, D. 15), at a rate of 7 cu. cm. per min. there shall distil at 760 mm. pressure: Not less than 60 cu. cm. at or below 90°, not less than 75 cu. cm. at or below 100°, not less than 90 cu. cm. at or below 120°. The flask shall be dry at a temperature not exceeding 125° (257° F.). The temperature recorded by the thermometer shall be corrected for emergent stem and barometric pressure, by means of the formulas given in the appendix. Residue. The amount of non-volatile residue remaining, when 20 cu. cm. of the material are evaporated, and subsequently heated for 2 hrs. on a water-bath at 100°, shall not exceed 0.01% by weight. Coloration with sulfuric acid. The coloration imparted to pure H<sub>2</sub>SO<sub>4</sub> (90% H<sub>2</sub>SO<sub>4</sub>), free from HNO<sub>3</sub>, when 90 cu. cm. of the material are shaken with 10 cu. cm. of the acid for 5 min., at ordinary temps., shall be not more than a light brown. Sulfur content. The total sulfur content shall not exceed 0.4% by weight. A convenient method for the estimation of S is given in the appendix. Freedom from acid and alkali. The material shall give no reaction for free acid or free alkali, when tested by the method described in the appendix. Freedom from hydrogen sulfide. The material shall give no reaction for H<sub>2</sub>S when tested by the method described in the appendix. Freezing point. The f. p. shall not be higher than 14° (7° F.) when determined by the method described in the appendix. The appendices give details of: Method of testing for color; temperature corrections for emergent stem and barometric pressures; methods of testing for S, free acid or free alkali, H<sub>2</sub>S, f. p.; and details of the standard distillation apps.

=> FILE CAPLUS

COST IN U.S. DOLLARS

SINCE FILE

TOTAL

ENTRY

SESSION

FULL ESTIMATED COST

204.52

208.09

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

SINCE FILE

TOTAL

ENTRY

SESSION

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L14 359 L13

=> FILE CAPLUS

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ENTRY	SESSION

FULL ESTIMATED COST

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DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)

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ENTRY	SESSION

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=> S L14 AND XYLENE

109467 XYLENE

7572 XYLENES

111379 XYLENE

(XYLENE OR XYLENES)

L15 2 L14 AND XYLENE

=> D 1-2 IBIB ABS

L15 ANSWER 1 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2004:467886 CAPLUS

DOCUMENT NUMBER: 141:38632

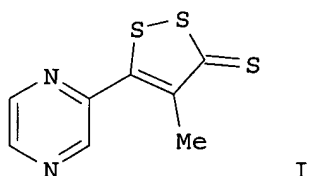
TITLE: Method for preparing oltipraz

INVENTOR(S): Kim, Jin-Wan; Choi, Kwang-Dong; Lim, Jee-Woong; Lee, Kwang-Hyeg; Lee, Sang-Ho

PATENT ASSIGNEE(S): CJ Corporation, S. Korea

SOURCE: PCT Int. Appl., 16 pp.  
 CODEN: PIXXD2  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004048369	A1	20040610	WO 2003-KR2552	20031125
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
KR 2004046274	A	20040605	KR 2002-74119	20021126
AU 2003284758	A1	20040618	AU 2003-284758	20031125
EP 1565460	A1	20050824	EP 2003-774311	20031125
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
CN 1717406	A	20060104	CN 2003-80104287	20031125
JP 2006511508	T	20060406	JP 2004-555112	20031125
US 2006106079	A1	20060518	US 2005-536492	20050525
PRIORITY APPLN. INFO.:			KR 2002-74119	A 20021126
			WO 2003-KR2552	W 20031125
OTHER SOURCE(S):		CASREACT 141:38632		
GI				



AB The method for preparing oltipraz I which includes reacting Me 2-methyl-3-(pyrazin-2-yl)-3-oxopropionate (II) with phosphorus pentasulfide in the presence of a mixture of toluene and xylene as solvent, followed by recrystn., is provided. The intermediate II was prepared by reacting Me pyrazine-2-carboxylate with Me propionate in the presence of tert-BuOK in THF.

REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L15 ANSWER 2 OF 2 CAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2002:256403 CAPLUS

DOCUMENT NUMBER: 136:278925

TITLE: Preparation of thionoesters, thiolactones, or dithiolethiones via a phosphorus pentasulfide, hexamethyldisiloxane, and sulfur composition.

INVENTOR(S): Curphey, Thomas J.

PATENT ASSIGNEE(S): Trustees of Dartmouth College, USA

SOURCE: PCT Int. Appl., 20 pp.

CODEN: PIXXD2

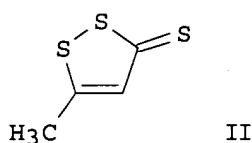
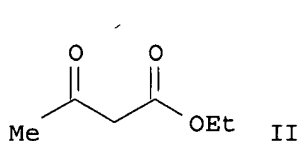
DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002026909	A1	20020404	WO 2001-US29937	20010925
W: CA, JP, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR				
US 2003176714	A1	20030918	US 2003-344014	20030509
US 7012148	B2	20060314		
PRIORITY APPLN. INFO.:			US 2000-236494P	P 20000929
			US 2001-313105P	P 20010817
			WO 2001-US29937	W 20010925
OTHER SOURCE(S):		CASREACT 136:278925		
GI				



AB A novel process is disclosed for the one-step thionation of esters, lactones, or 3-oxoesters. For example, a suspension of phosphorus pentasulfide (15 mmol), sulfur (25 mmol), and hexamethyldisiloxane (75 mmol) in dry xylene (50 mL) was refluxed under an atmospheric of dry argon for 5 min [Caution: fume hood - evolution of hydrogen sulfide (H<sub>2</sub>S) and odor]. Et acetoacetate (I, 25 mmol) was added over a 3 min period and the mixture then refluxed for 1 h. The resulting red-brown solution was cooled to 0°C and finely powdered K<sub>2</sub>CO<sub>3</sub> (60 mmol) was added, followed by the dropwise addition of water (10 mL) [Caution: gas evolution], stirring in acetone, extraction, and evaporation. The oil was dissolved in toluene, filtered through a short column of silica gel and activated carbon, and the solvent removed in a vacuum. The oil was recrystd. from carbon tetrachloride at -15 0 °C to give dithiolethione II as orange crystals in 80% yield (mp 33.5-34.5 °C). Use of this reagent for thionation of 3-oxoesters significantly increased the yield of the dithiolethione over existing methods and simplified the production of the final product. A direct comparison was made of the method of the present invention for thionation and dithiolethione synthesis with the method employing Lawesson's reagent. In all trials examined, e.g., 14 examples, the method of the present invention was more effective than the use of Lawesson's reagent. Further, these methods have the advantage that reagent-derived byproducts may be removed by a simple hydrolytic workup or by filtration through silica gel, rather than by chromatog., as required by Lawesson's reagent.

REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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---Logging off of STN---

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